A Study Into Life Modelling for Elastomeric Tubes

Thesis

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A Study into Life Modelling for Elastomeric Tubes

Thesis presented in candidature for the award of the degree of Doctor of Philosophy

Nancy Ashburn

BSc Hons, MEng

Faculty of Mathematics, Computing & Technology Materials Engineering

The Open University

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ABSTRACT

The performance of a tube within a peristaltic pump is as a result of complex interactions between material, the process used to produce the tube, the pump it is in and the environment to which it is subjected. This research examines a possible methodology for the development of life modeling for elastomeric tubes used in peristaltic pumps. It is shown that predicting life within a peristaltic pump is a complex process but that the ability to predict the life cycle of the tube can be enhanced through material and process understanding and innovation. A systematic approach is detailed for the analysis of a tube life cycle from raw material through to failure. This tube life cycle could be used as the basis of a life modeling algorithm, a conceptual design for this is suggested.

Using two materials highlighted as important to the peristaltic pump industry, detailed tube life analysis is carried out to show how the methodology can be implemented. The approach suggests key indicators that can be used to identify material characteristics which influence the life of a tube. This is shown for the two materials studied and how they differ from material to material.

Extrusion methods for each of the materials is analysed in some detail and changes to, or controls for, the extrusion process to produce tubing are put forward. It is suggested that this will produce tubing which will perform more consistently within a peristaltic pump. This consistency of performance is put forward as a key facilitator for life modeling.

Environmental factors which influence life are identified; with system pressure and temperature being the most influential on life. The way all the factors identified interact is discussed. From the identification of these factors appropriate sensor inputs are put forward which will enable them to be monitored and used within an algorithm.

Contents

GLOSSARY

- HTV: High temperature vulcanised: materials that require a high temperature by which to activate the curing reaction.

- RTV: Room temperature vulcanised: materials that require a room temperature, circa 23°C by which to activate the curing reaction.

- Shore A hardness: A measure of the hardness of an elastomer. Based on the depth of the indentation by a standard size and shape impacting gauge. The Shore A scale range from 30 to 95 points, the higher the points the harder material. This Durometer scale was defined by Albert.F.Shore.

- PPS: Polyphenylene Sulfide

- ABS: Acrylonitrile Butadiene Styrene

- Solubility Parameter: A parameter used in predicting the solubility of one material with another, materials with similar solubility parameters are likely to be miscible.

- Qd: Flow drop

- kGy: KiloGray: A dose measurement unit for the amount of radiation absorbed by a material

- PDMS: PolyDiMethylSiloxane

- Transfer Pressure: A system pressure of between 0 and 0.5 bar

1 INTRODUCTION

1.1 Background

A large number of industries around the world use peristaltic pumps. They include the water treatment industry, where peristaltic pumps are used to meter chemicals, such as sodium hypochlorite, to maintain water cleanliness; the food industry, where for example they dispense fruit syrup into the yoghurts we see at supermarkets; in the dosing of dye into paper at paper mills; and to dose vaccines into the food stock of animals. Their use extends to the dispensing of sugars and yeast into beer at breweries and dosing chemicals which maintain pH control at mining installations, to name just a few applications for these pumps.

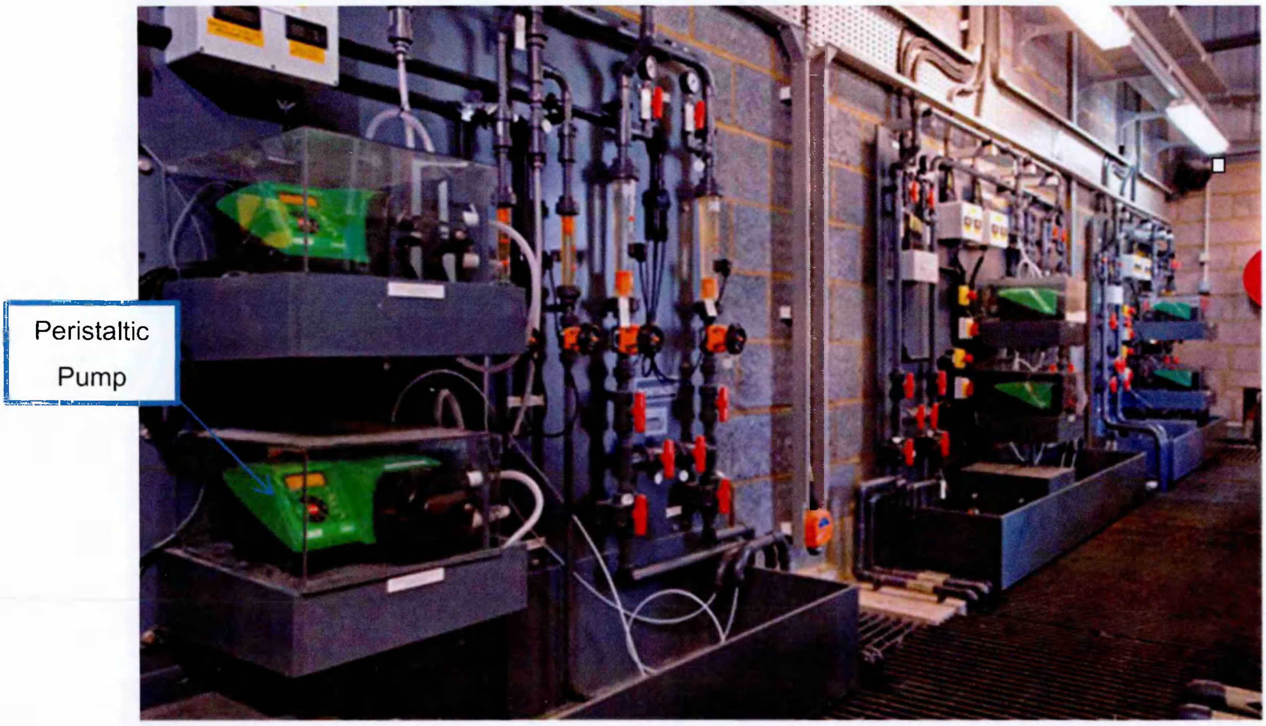


Figure 1 - Peristaltic pumps pumping sodium hypochlorite at a large waste water treatment plant in the UK (1)

Peristaltic pumps in their simplest terms work on the peristalsis process seen in the human digestive system, where food is pushed along by consecutive constricting muscles, in a wave format.

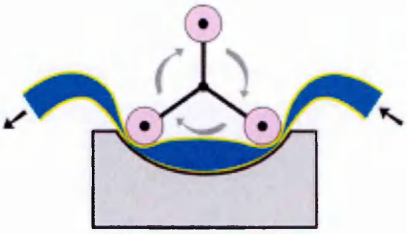


Figure 2 - The peristaltic action (2)

In a peristaltic pump the movement of the fluid is achieved by occluding (constricting) a tube between a fixed track and a roller or shoe, located on a rotating rotor.

In this way the fluid is drawn into the pump and then expelled. Nothing but the tube touches the fluid.

The level of occlusion, (o) (defined as how much the tube is compressed) is governed by the wall thickness (w) of the tube and the minimum gap (g) between the roller and the track.

$$o = 2 \times w - g \quad \text{(Equation 1)}$$

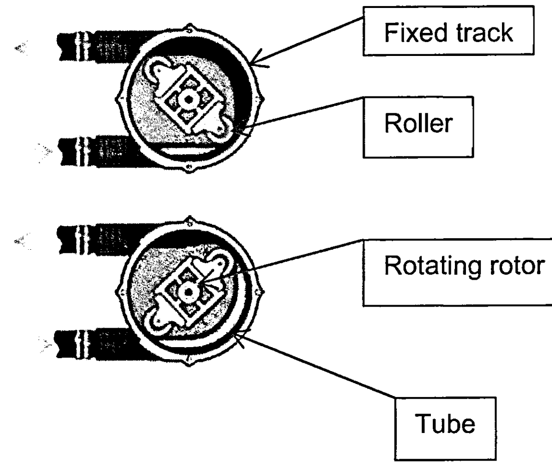


Figure 3- A simple rotary peristaltic pump (6)

The complete closure of the tube when it is squeezed in this way gives the pump a positive displacement action, whereby the flow rate is constant regardless of the system pressure, or head, in which the peristaltic pump is placed. It thus forms part of the positive displacement pump family.

1.1.1 Positive Displacement Pumps

The positive displacement pump family is made up of a number of different pump types:

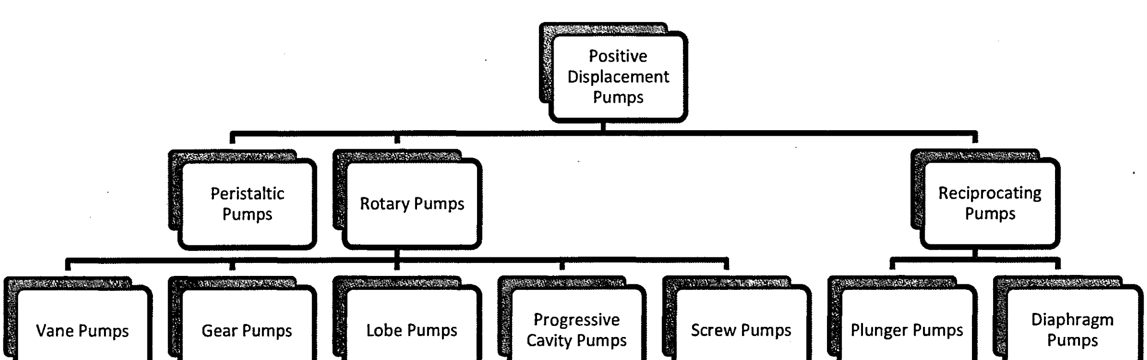


Figure 4 - Positive displacement pump classification diagram

The relative market share within the positive displacement pump sector for peristaltic pumps is small, but they are the fastest growing type of pump within the positive displacement pump market (3).

When compared to others within the positive displacement pump family, peristaltic pumps show some obvious differences in performance characteristics. One example pertains to diaphragm pumps. These are unable to pump entrained air as found in sodium hypochlorite applications, such as those seen in Figure 1.

The sodium hypochlorite naturally decomposes over time releasing a variety of gaseous by-products. This gas or even a pocket of air in the line will often cause a diaphragm pump to lose its' pumping action even though the pump is still running. This phenomenon is called 'vapour locking'. When this occurs, an operator must bleed the air from the line and re-prime the pump. This is not necessary with peristaltic pumps, as the pump will pump the pocket of air or gas as efficiently as it will pump the sodium hypochlorite.

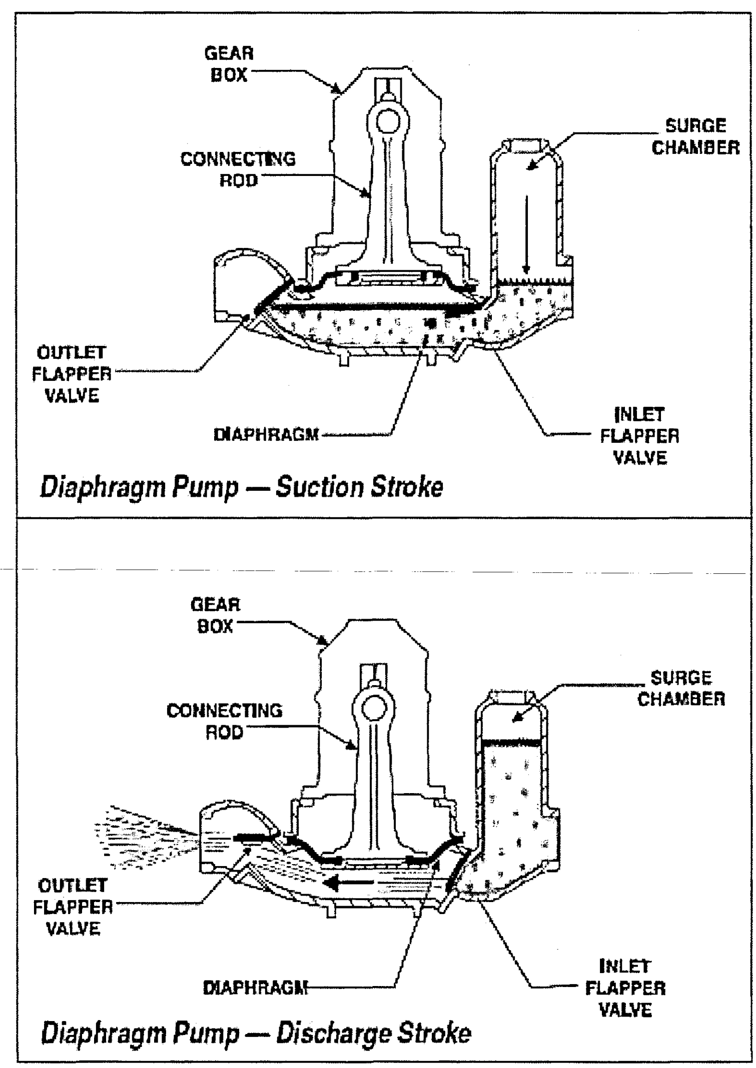


Figure 5 - Diaphragm pumps (4)

The characteristics displayed by the different positive displacement pump types are highlighted in the

Table 1; along with simplified images of some these pump types:

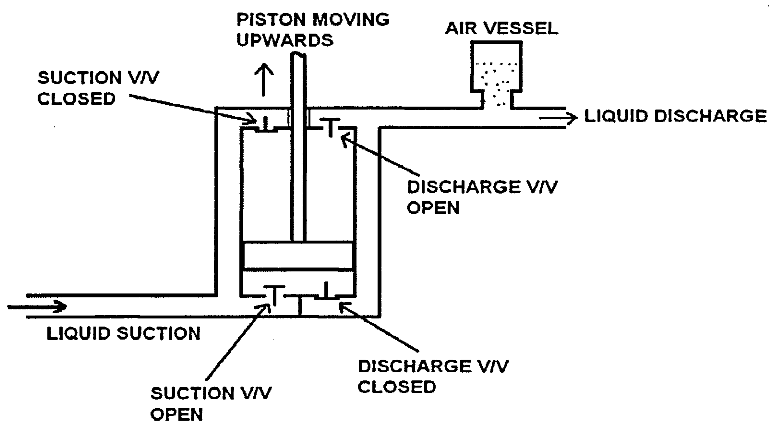


Figure 6 - Plunger or Piston type pump (5)

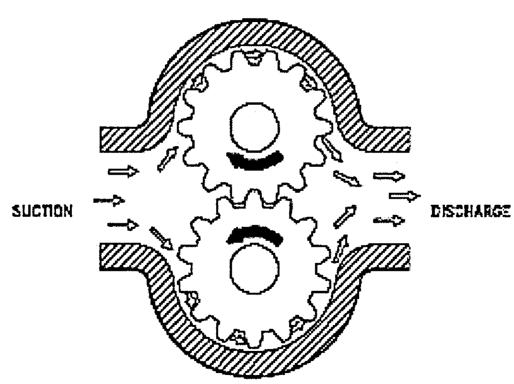


Figure 7 - Gear pump (6)

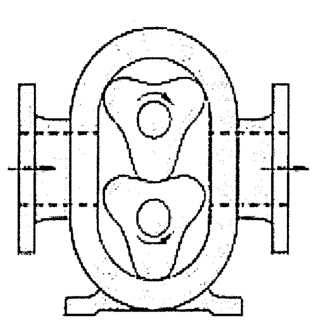


Figure 8 - Lobe pump (7)

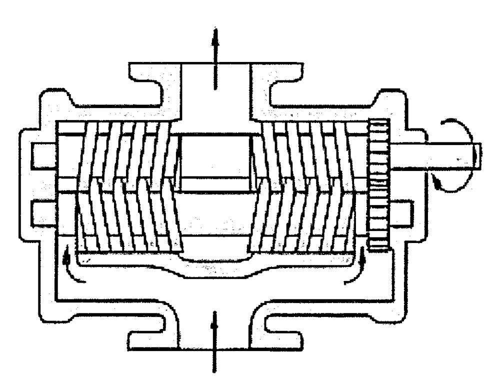


Figure 9 - Screw pump (8)

PUMP TYPE	PROS	CONS
Progressive Cavity Pumps	Reliable movement of viscous fluids and slurries.	No complete sealing Abrasive fluids will shorten life
Vane Pumps	Can be used as a variable displacement pump. Can cope with viscous fluids	No complete sealing High wear
Gear Pumps – see Figure 7	Can pump highly viscous fluids at high pressures.	Gear fitment critical – abrasive fluids will shorten life When used with viscous fluids , speed must be reduced considerably so pump-head can be filled fast enough
Screw Pumps – see Figure 9	Can pump highly viscous fluids at high pressures.	Clearances are minimal so abrasive fluids can't be used.
Lobe Pumps - see Figure 8	High capacity Low pulsation – gentle performance on fluid being pumped	Clearances are minimal so abrasive fluids can't be used.
Diaphragm Pumps – see Figure 5	Can be low cost Complete sealing achieved Handle viscous fluids	Can't pump air Need flooded suction to prime Large number of replaceable parts in the pump head
Plunger Pumps – see Figure 6	Can pump high viscosity	Has a dead band when the system pressure reaches a certain level, the pump motor stops causing leak-back of the pump medium
Peristaltic Pumps	Pump mechanism separate to the pump medium Does not need flooded suction to prime. Only need to change the tube when failure occurs Can cope with abrasive and viscous fluids	Tube failure is unpredictable Very high pressure applications can be difficult to deal with as reliant on elastomer technology

Table 1 - Pros and Cons of different displacement pump types

1.1.2 Peristaltic Pumps

The peristaltic pump consists of four key parts: a drive, a pump head, the tube and the medium being pumped.

The drive itself is made up from three basic sub-systems: the power supply unit, a motor system and the human-machine interface, all enclosed within a case which provides protection from the environment and a means of assembly.

The pump head has two distinct parts: a fixed track and the rotating rotor, which are attached to the case and drive motor system respectively.

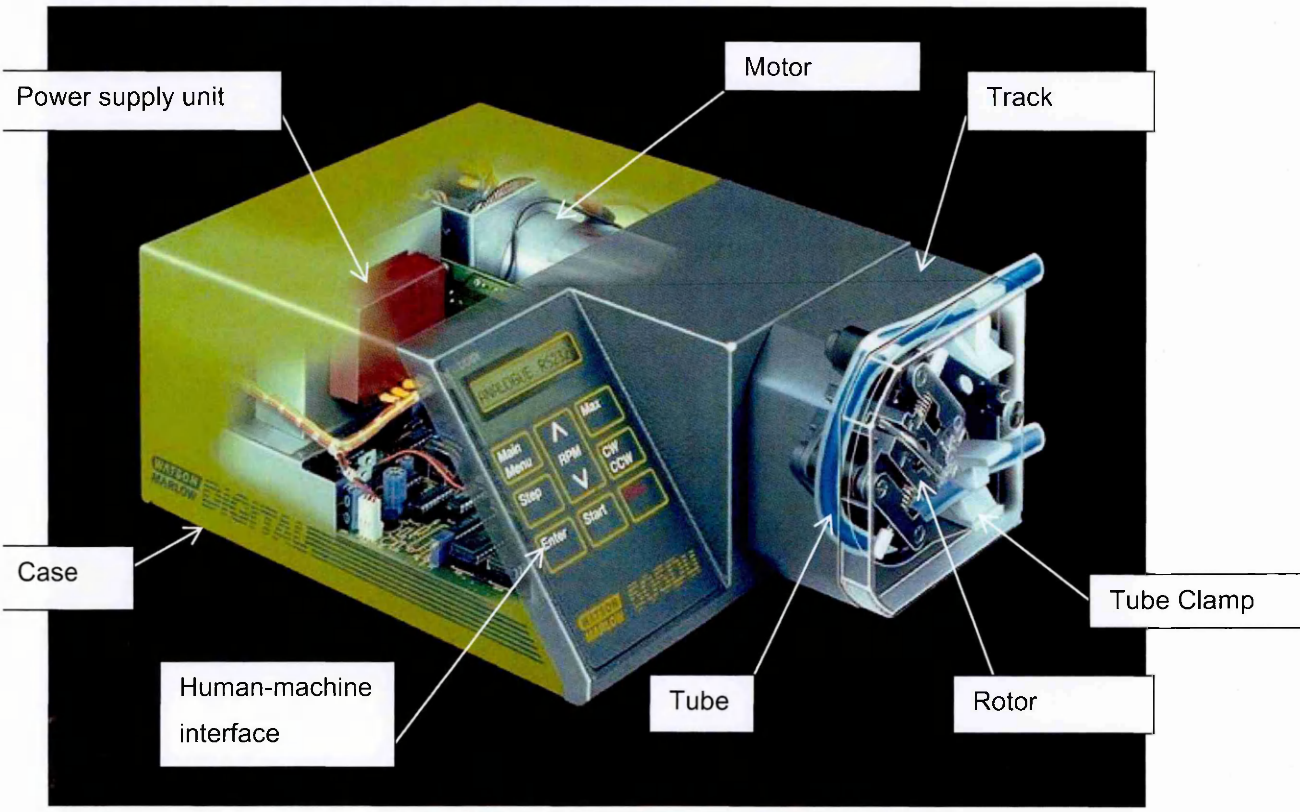


Figure 10 – The key parts of a peristaltic pump

The pump heads vary in a number of ways:

- the track material
- the track geometry
- the rotor configuration, whether it has shoes or rollers i.e. whether it is fixed or sprung
- the rotor material
- the tube clamping arrangement, whether the pump head can take continuous lengths of tube or whether it is designed for fixed lengths held within a element assembly.

Each pump head is designed to accommodate a certain range of tube sizes and materials and to deliver a range of flows and pressures.

The flow rates of peristaltic pumps are dependent on the actuation frequency (the RPM x the number of actuators, i.e. rollers or shoes), and the volume displaced during the occlusion, often termed the pillow volume. If complete occlusion is assumed then the maximum pillow volume for each tube size can be estimated from its internal diameter and the length of the tube being actuated.

$$\mathrm{Q = rpm \times no. of\ roller \times pillow\ volume} \qquad \text{(Equation 2)}$$

Incomplete occlusion, stretching of the tube material, and the volume occupied by the actuators themselves can cause the flow rate to be lower than the estimated maximum pillow volume. So for a particular peristaltic pump each tube size fitted will be capable of a certain flow rate at a certain rotor speed depending on its bore and the material from which it is made.

All tubes are selected by the end user to give a certain flow rate in a certain pumping environment, where the following environmental conditions are all considered:

- temperature, both ambient and of the medium pumped
- system pressure
- the medium being pumped – chemical compatibility, leachability issues, etc
- the performance of the tube required – in terms of accuracy of doses, life, etc
- any sterilisation or cleaning regimes that may be employed

For all peristaltic pumps the tube is the key to its performance, therefore all peristaltic pump users rely on an elastomeric tube for their application to succeed.

1.2 Tube Materials

Since the first peristaltic pump was invented in 1935 by Dr Michael DeBakey (9) they have been typically used to pump aggressive or clean / sterile fluids where contamination with exposed pump components seen in other pump types means that the use of the tube within the peristaltic pumps offers the user the separation they need.

For a peristaltic pump the tube material choice is vital, the tube within a peristaltic pump must open back to its natural state after the passing of the roller, as it is this restitution which draws in the next package of liquid into the pump. This repetitive peristaltic action implies the need for a peristaltic tube to display good fatigue resistance.

Studies specific to peristaltic pumps (10) (11) have highlighted that the stresses imposed by the peristaltic action are both non-linear and extremely complex. However, finite element analysis has been used with some success within these studies to highlight the high stress areas and this can be used to help determine where likely failure points will occur.

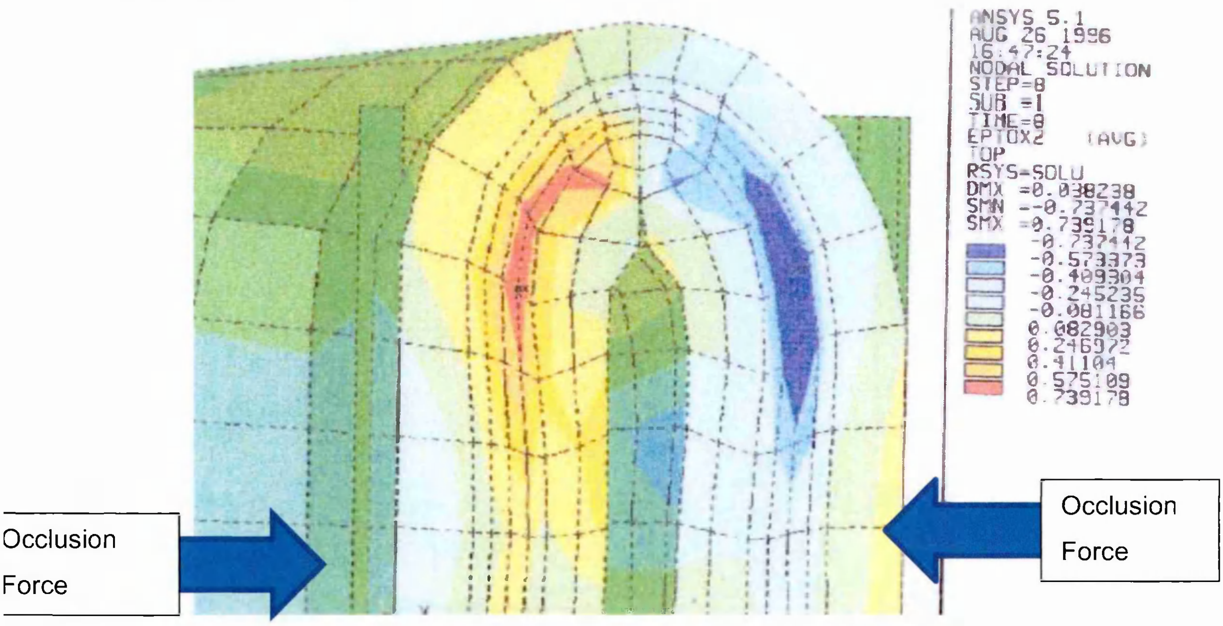


Figure 11 - Shear strain in x'z direction, dark areas indicate maxima (11)

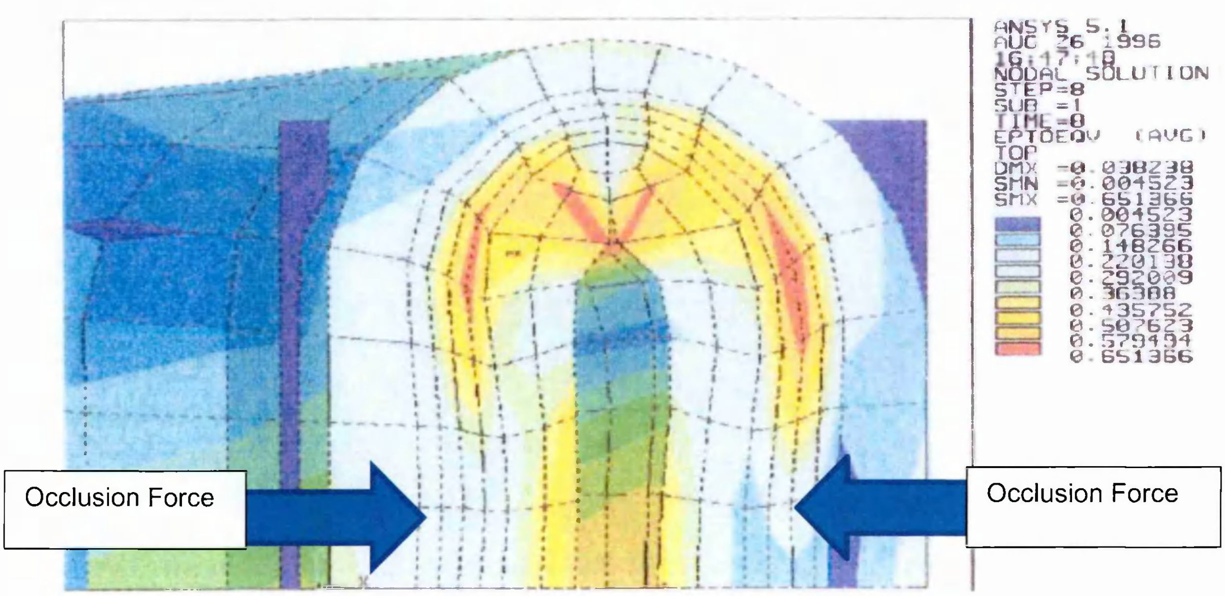


Figure 12 - Tensile strain in a cross section of tube, displacement by shoe (11)

However the complexity of the stress model means it can be difficult to understand, unless one is an expert in finite element analysis, so although it can be used to broadly show where high stress areas are, the study (11) showed that the model could not be realistically applied to real tubes, thus suggesting that the overall stress pattern is not completely understood. The study also suggested that the specific properties needed in a material cannot always be determined through the use of standard test regimes and results.

One study (12) looked at the distribution of shear stress in the tube wall in a two roller based peristaltic pump. When the rollers were positioned with an angle θ = 216˚ or 144˚ only one roller occluded the tube. It found that when the rollers were position θ = 180˚, see Figure 13, the position when both rollers are almost occluding the tube, that there is a sharp peak in shear stress within the tube wall.

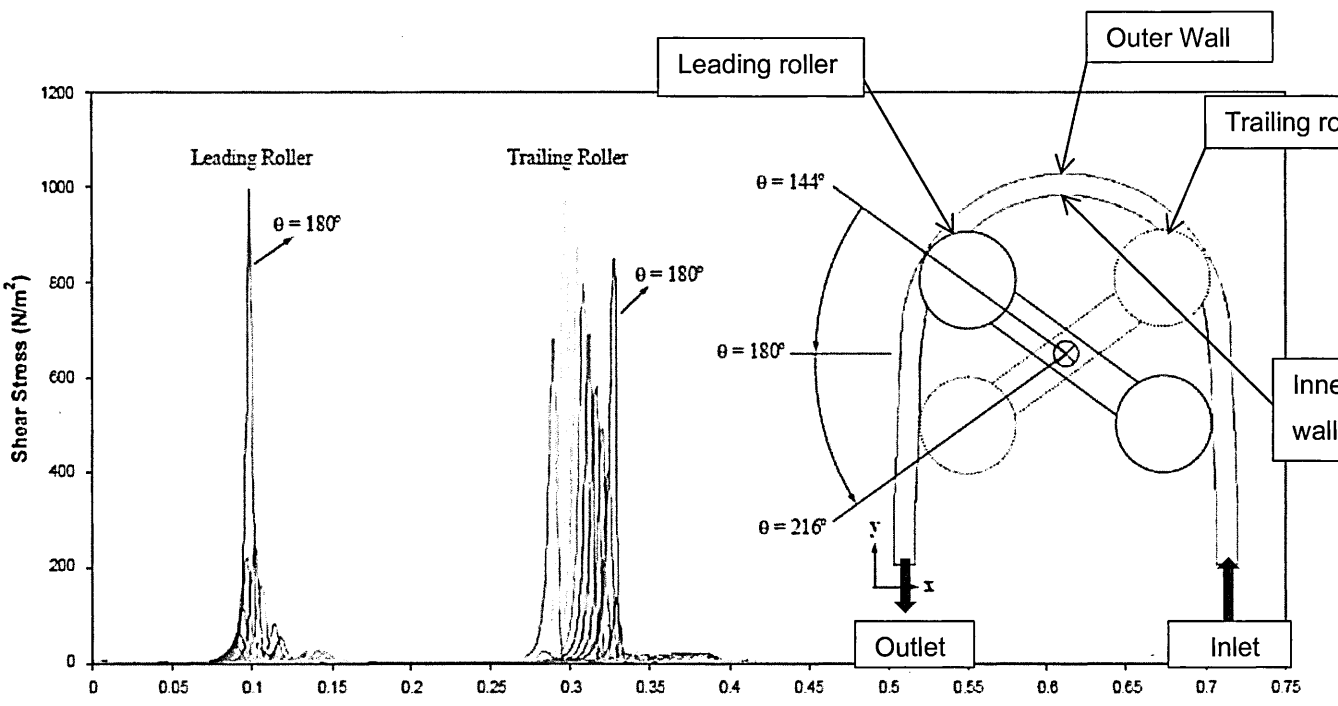


Figure 13 - Wall shear stress distribution along the arc length of the inner wall (12)

As the trailing roller comes into contact the volume of tube decreases, the fluid is incompressible and thus the pressure between the rollers becomes very high, the fluid is therefore driven through the narrow gap between the inner and outer wall very fast. This study suggests that this high velocity gradient gives rise to the sharp peak in shear stress. It was also found that there were higher shear stresses at the inner wall than at the outer wall, attributed to the velocity profile being more skewed towards the inner wall due to the centrifugal force, giving rise to a higher gradient. This study highlighted the fact that the outlet velocity has been shown to be much higher than the inlet velocity with corresponding higher shear stress levels. This corresponds to the stress analysis carried out using finite element analysis in previous studies (11).

The tube material must offer not just the mechanical properties required by the peristaltic mechanism, but also the inert, clean and sterile properties needed for the medium they are pumping. There are a variety of tube materials ranging from natural rubbers, to silicone rubbers; from composite and reinforced materials to thermoplastic elastomers - thermoplastic elastomers being copolymers or physical mixtures of materials which have both thermoplastic and elastomeric properties, examples being styrenic block copolymers and thermoplastic polyurethanes.

However a number of these materials require greater consideration due to their relative importance within the peristaltic pump industry where they have come to the fore due to either their inert, clean and sterile properties, such as with silicone rubber; or for their ability to deal with more aggressive and industrial applications, such as the thermoplastic elastomer, Santoprene ® .

1.2.1 Silicone Rubber

Silicone rubber is an elastomer (rubber like material) composed of a silicone polymer, polydimethylsiloxane, with a repeat unit [$-Si(CH_3)_2O-$]. The material first became available in the 1940's through work by Dow Corning and then Wacker Chemie, with the first mention of silicone rubber tubing within the same decade. It has been considerably developed to optimise its properties.

Although a highly inert material with good resistance to extreme temperatures; dimethylsiloxanes can withstand temperatures above 200˚C in the absence of oxygen (13) and are typically quoted for use between -55˚C and 300˚C, its fatigue strength when unfilled is lower then many other rubber materials, however the typical ultimate tensile strength of a silica filled material with a hardness of 60 Shore A is 8.96MPa. With a T_m near -40˚C and a T_g of about -120˚C the low temperature properties are also worth noting.

Of most use within the peristaltic pump industry are the high temperature vulcanised, HTV, silicone rubbers. These are divided into solid rubbers and liquid silicone rubbers. Both materials consist of polydimethylsiloxane with reactive vinyl groups, finely dispersed silica and a suitable crosslinking agent. Solid rubbers are higher molecular weight polymers than the liquid silicone rubbers, generally having molecular weights of between 400,000 and 600,000 g/mol (13).

The crosslinking of the polymer is achieved through the use of catalysts such as platinum or peroxide-based catalysts and thermal curing. It can also be crosslinked through electron beam crosslinking (14), however this has not yet become a fully commercialised process. Within the manufacture of peristaltic pump tubing it is the use of platinum based catalysts that have come to the fore commercially for high temperature vulcanised, HTV, solid silicone rubbers in recent years, this arose out of concerns about the peroxide catalyst byproducts in the biomedical sector of the sanitary market. For the liquid silicone rubbers platinum catalyst have been used for some time (13).

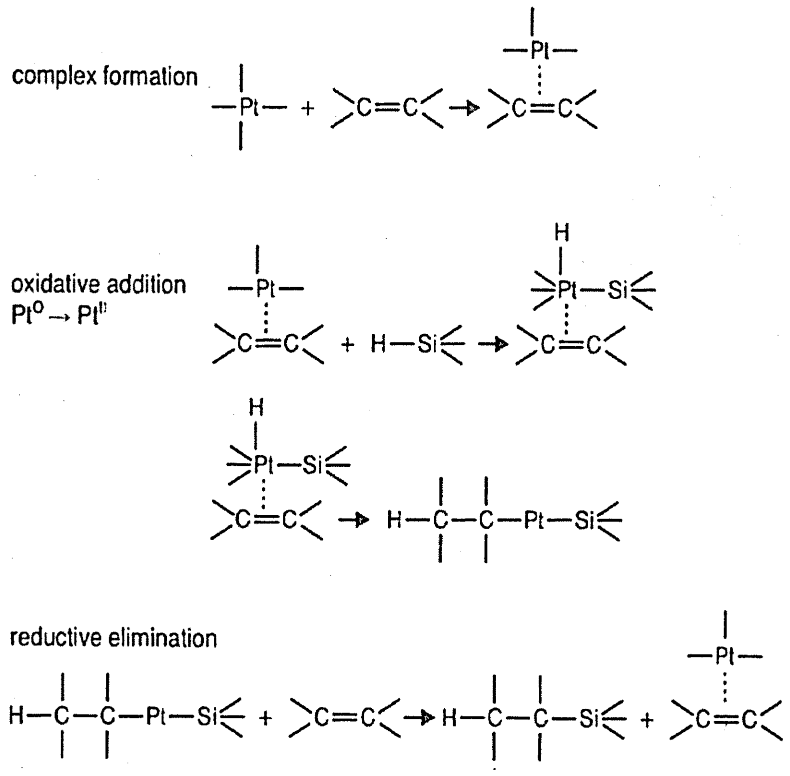


Figure 14 - Platinum catalyst addition curing of silicone rubber (13)

The cross-linking process forms a network structure which others have found can be characterised by a number of topological parameters, such as length of network strands, functionality of crosslinks and amounts of entanglements, dangling chains and loops. However, the molecular understanding of the network topology-mechanical property relationships still remain incompletely understood (15).

Crosslinked pure silicone rubber shows a low tensile strength due to their low intermolecular interactions, only with the additions of reinforcing fillers can high strength silicone rubber be obtained. The reinforcing potential of particle addition into elastomers is well acknowledged (16) (17) (18) (19), particularly the use of carbon black and silica types within a great variety of rubber products, giving increased stiffness, modulus, rupture energy, tear strength, tensile strength, cracking resistance, fatigue resistance and abrasion resistance.

However, within the pump industry it is the use of silica filler in silicone rubber which is most prevalent due to its optical clarity and the fact that the addition of silica filler to silicone rubber further enhances the reinforcing qualities by promoting the chemical interaction between the silica reinforcement and the PDMS material.

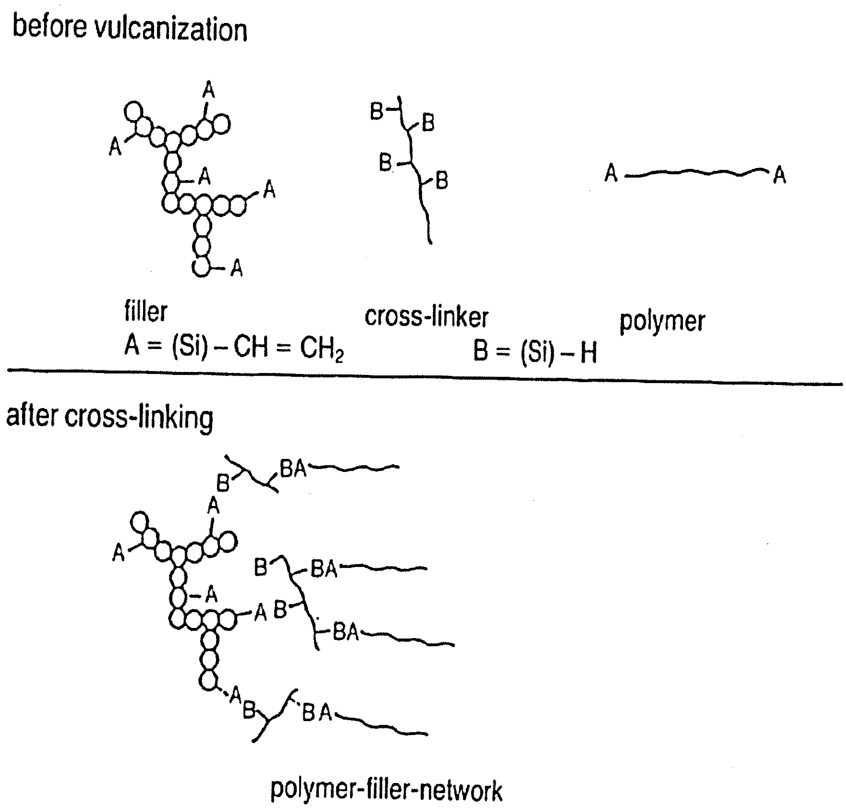


Figure 15 - Network formation during the cross-linking process (13)

Within the silicone rubber industry the effectiveness of silica filler has been studied in detail (20) (21) (22) (23) (24).

It has been found that the effectiveness of the filler is dependent on a number of key characteristics:

- The amount of filler added – volume fraction of the particles.
- The size of the filler particle, both as a primary particle and at an aggregate level.
- The shape of the filler particle – spatial morphology of both the primary particle and any aggregates formed.
- The strength of the polymer-filler interaction – determined by the functional groups on the particle surface.
- The basic chemical network of the polymer.

Both particle-to-particle and particle-to-polymer interactions occur in the formation of the silicone network structure. Research suggests that filler size, filler loading (see Figure 16), and distribution most heavily affect the amount of polymer interaction, whilst the surface structure and chemistry of the particle dictate the intensity of the interaction at the particle-to-polymer interface (25).

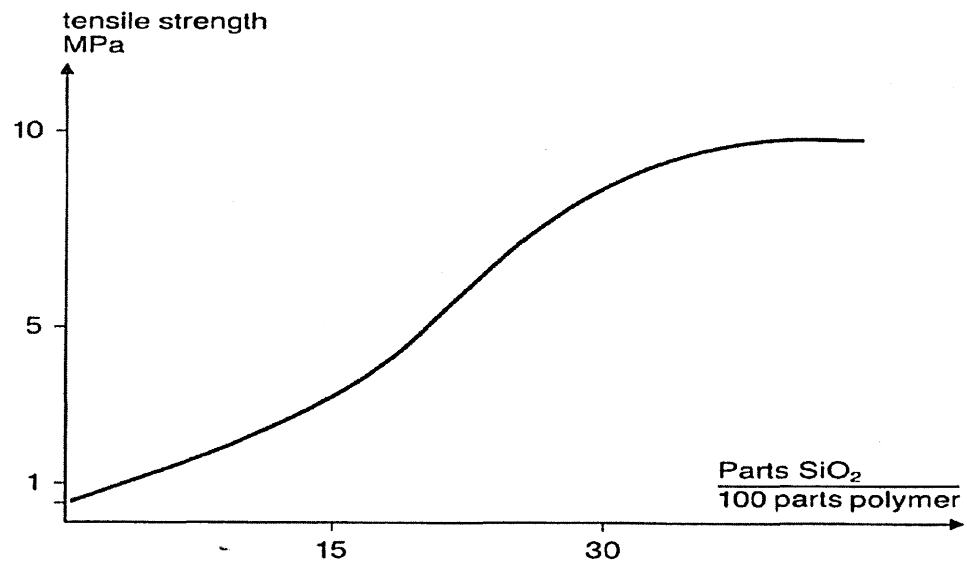


Figure 16 - Relationship between tensile strength and amount of fumed silica filler- filler surface area 150 to 400m²/g (13)

The particle-to-polymer interaction takes place when the oxygen atoms of the polymer molecules form hydrogen bonds to the surface silanol groups of the particle, forming a bound rubber layer, the thickness of which depends on the polymer molecular weight or chain length.

Filler particles are usually blended into the polymers before the cross-linking reaction, they tend to agglomerate due to the hydrogen bonding between particles, leading to a rather inhomogeneous material, see Figure 17. In order to process the material chemical modification of the surface of the silica particles can be used to tailor the reactivity of the silica. These processing aids, such as hexaalkyldisilazanes (13) produce a hydrophobic silica surface reducing the thickening effect of the filler on the silicone rubber when being processed; they are particularly effective for liquid silicone rubbers.

Solid rubbers use short chained polysiloxanediols as a processing aid, these have a reduced hydrophobic effect which allows the rubber to maintain its rigidity while being processed. Other factors that can be used to tailor the reactivity of the silica are the particle specific surface area and shape; to increase dispersion and reduce agglomerate formation. This must be tailored with the need for enhanced interaction between the filler and the polymer which allows better transfer of stress from the polymer matrix to the filler giving a strong network structure.

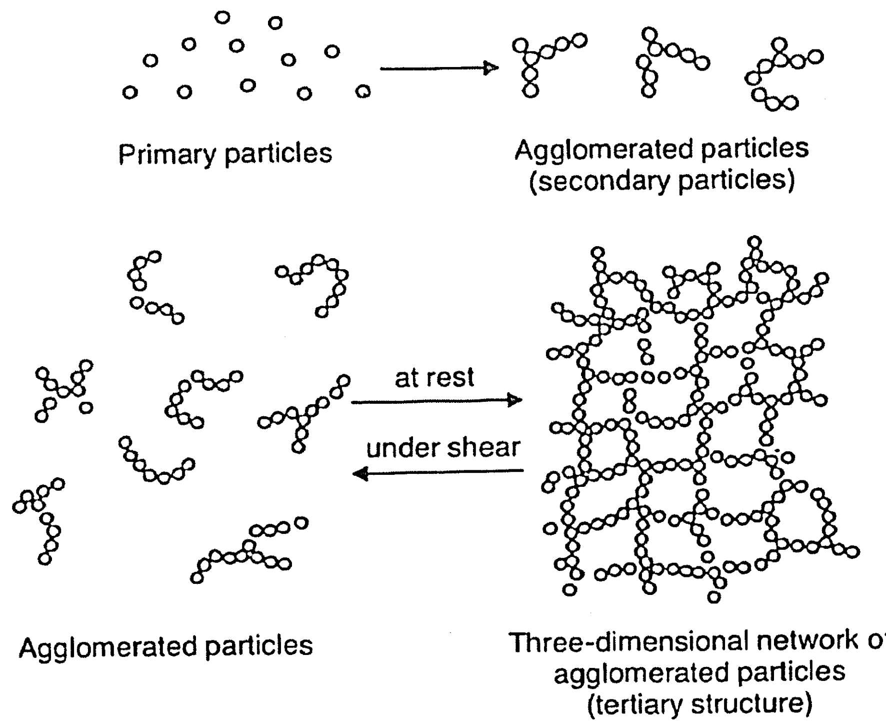


Figure 17 - Interaction between silica particles showing how small primary particles can form larger agglomerated structures if surface modification is not utilised (13)

1.2.2 EPDM & PP Blends

Thermoplastic elastomers have been widely used as the materials in automobile, industrial and electrical products for a number of years.

Within the thermoplastic elastomer family of materials are the thermoplastic (dynamic) vulcanizates (TPVs). These are produced by dynamic vulcanization (crosslinking, curing) of compatibilized blends made up of thermoplastic resins and cross-linkable rubbers. The dynamic vulcanisation process is aimed at selectively cross-linking the dispersed rubber phase, without extensive propagation of the cross-linking reaction into the PP phase (26).

One of the most common commercial TPVs is the blend made up from polypropylene (PP) and vulcanized ethylene/propylene/diene rubber (EPDM) of which the Santoprene® family of products by Advanced Elastomer Systems, a subsidiary of ExxonMobil, is one. This material is also classed as an olefinic thermoplastic elastomer. It typically demonstrates a melt endotherm from 120˚C to 164˚C for an 80 Shore A material, with a peak at 155˚C (27). Its operating temperature range when used for pump tubing is 5˚C to 80˚.

$-CH_2-CH(CH_3)-$ & $-(CH_2- CH_2=C(CH_3)-CH_2-CH(CH_2-CH=CH-CH_3)-CH_2)-$

Figure 18 – Repeat Units for PP and EPDM

The PP/EPDM weight ratio can be varied in a wide range to adjust several TPE properties like hardness, tensile modulus, elongation at break, compression set, oil resistance and others. For example the hardness of the material is in the main governed by the PP content, which increases hardness. An EPDM/PP blend with a hardness of about 94 Shore A will show a tensile strength of 15.6MPa and an elongation at break of 560%, see appendix 9.2.

The EPDM / PP blends used to manufacture tubing used in pumps come in a range of hardness.
A number of detailed studies have shown that the optimisation of the properties of these blends is achieved by careful control of their morphology, particularly the dispersion of the elastomer into the thermoplastic phase and the size of the elastomeric particles (28) (29).

There are also a number of other additives within these materials which are used to tailor their properties, oil, fillers, pigments and stabilizers etc. Oils are used to lower hardness and thus improve the processing of the material; however they have also been shown to affect the thermal and mechanical properties of the blend and its rate of elastic recovery, lowering the temperatures of the glass transition temperature (T_g) of the EPDM and PP phases and the melting temperature (T_m) of the crystalline PP phase (26). The oil content shows preferential position in the EPDM phase of the material.

Fillers are commonly added to increase stiffness, improve dimensional stability and lower the cost of compounds.
However, it has been found with certain fillers within these blends, such as SiO_2, that efficient filler dispersion is crucial to avoid filler aggregation which can actually decrease impact strength (30). It has also been found that as with other reinforcing mechanisms the size and shape of the particles influence the effectiveness of the filler. Filler particles of a nanoscopic size in EPDM have been shown to form a filler network with larger reinforcement efficiency than fillers at a micron size (31); it has also been shown that the surface treatment of these fillers is important in the formation of an effective network structure (32). Increasingly polymer blending is becoming more sophisticated to achieve the best combination of mechanical properties (33) (34).

1.3 Tube Failure

Tube failure to the user has two major meanings:

1. Change in performance caused by a change in the tubes' mechanical properties
2. Failure of the tube due to fracture of the tube wall

In the first case for example this could be the point at which the pump head flow drops beyond the point where the pump is matching the flow requirement it has been set. A user is running a tube at a set rpm to achieve a flow of 100ml/minute: the tube material undergoes permanent deformation caused by the cyclic stresses applied to it; this means the tube is no longer able to maintain a flow of 100ml/minute at the same rpm, see Equation 2:

$$Q = \text{rpm} \times \text{no. of roller} \times \text{pillow volume} \qquad \text{(Equation 2).}$$

Many users can compensate for this type of failure within their system by increasing the speed of the drive to produce greater flow, in the case above by increasing the pump speed, so the flow returns to 100ml/minute.

The tube within a peristaltic pump can show changes in performance and signs of fatigue very early in its life, under repeated forces imposed on the tube, through the peristaltic action. The first thing noted is the permanent deformation that occurs due to the compression set or creep of the material. This results in the circular tube taking on an ovoid set. The level of compression set and how quickly it occurs is related to the occlusion level, the speed of rotor, the system pressure and the temperature. This mechanism is responsible for the reduction in the material's restitution characteristics, leading to a change in the tubes performance over time, particularly flow rate capability.

The tube failure mode depends on the tube material. In the EPDM/PP blends it has been observed that crack propagation occurs through the longitudinal crease, which builds up due to the repetitive compressive action on the tube. By sectioning a failed tube this cracking on the inner bore can be observed post-failure, see Figure 19, and it can be seen that the crack propagation is clearly linked to the highest stress area as identified in a previous FEA study (11). This failure mode is by far the most prevalent for EPDM/PP tubing within peristaltic pumps and is therefore considered typical. Other non-typical EPDM/PP failures which occur much more rarely are discussed later in chapter 3, but they include mechanical wear on the outer surface of the tube and environmental stress cracking due to a combination of the peristaltic action and chemical attack by the medium being pumped.

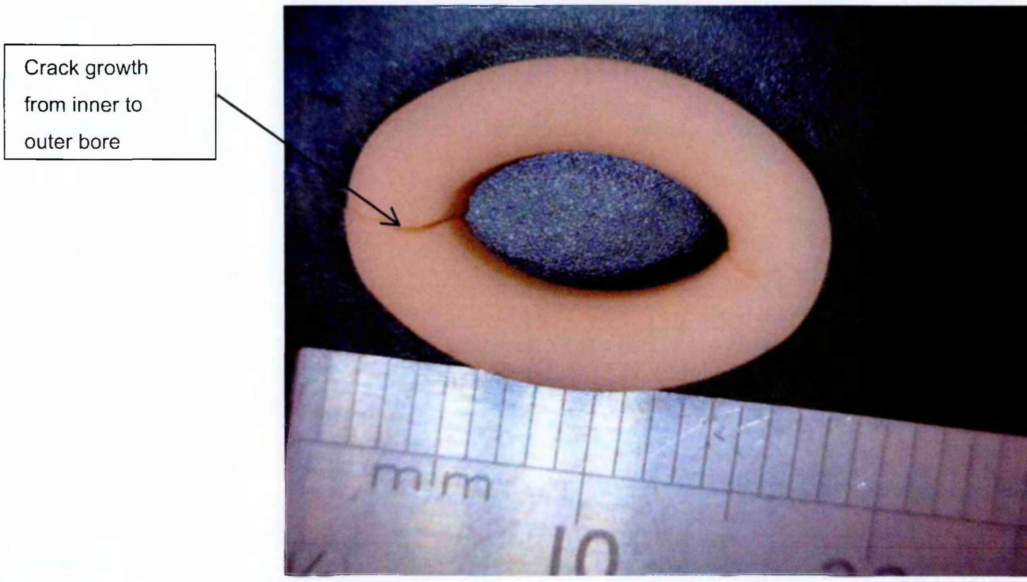


Figure 19 – A section through EPDM/PP tubing showing crack propagation on the inner bore

For silicone materials compression set and creep occurs as for the EPDM/PP blends, resulting in an ovoid compression set of the tube, see Figure 20, along with the longitudinal cracking as seen with the EPDM/PP material. However, in silicone rubber tubes in addition to longitudinal cracking, cracks in a direction parallel to the occlusion force are observed occurring in multiple sites along the tube, see Figure 21. It is important to note that in the majority of cases the fracture site, where the crack has broken through from the inner bore to the outer bore, occurs at the outlet end of the tube, which is the highest stress area. In addition, crazing and shear damage has been observed and are discussed more fully in chapter 3.

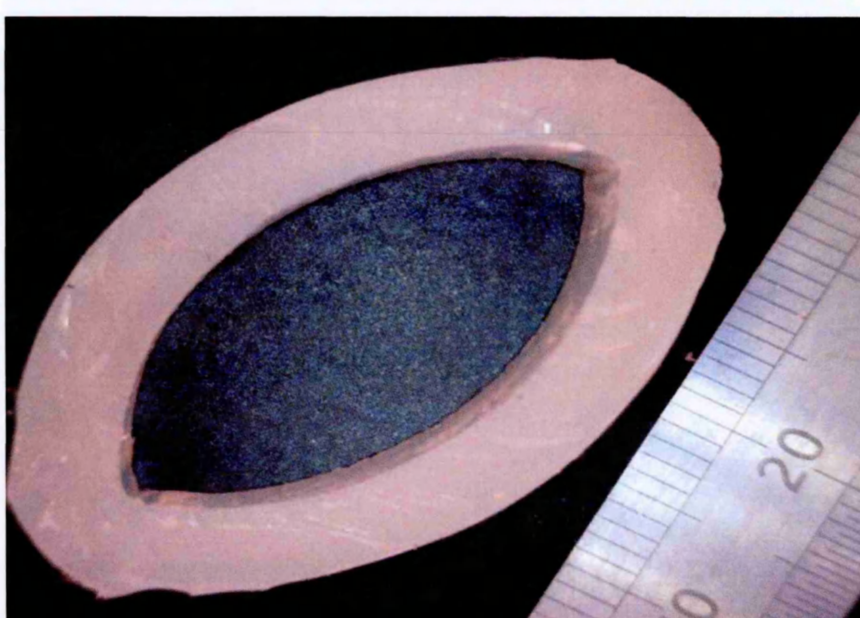


Figure 20 – A section through a Silicone rubber tubing showing compression set to the ovoid shape after just 4 hrs. of pumping

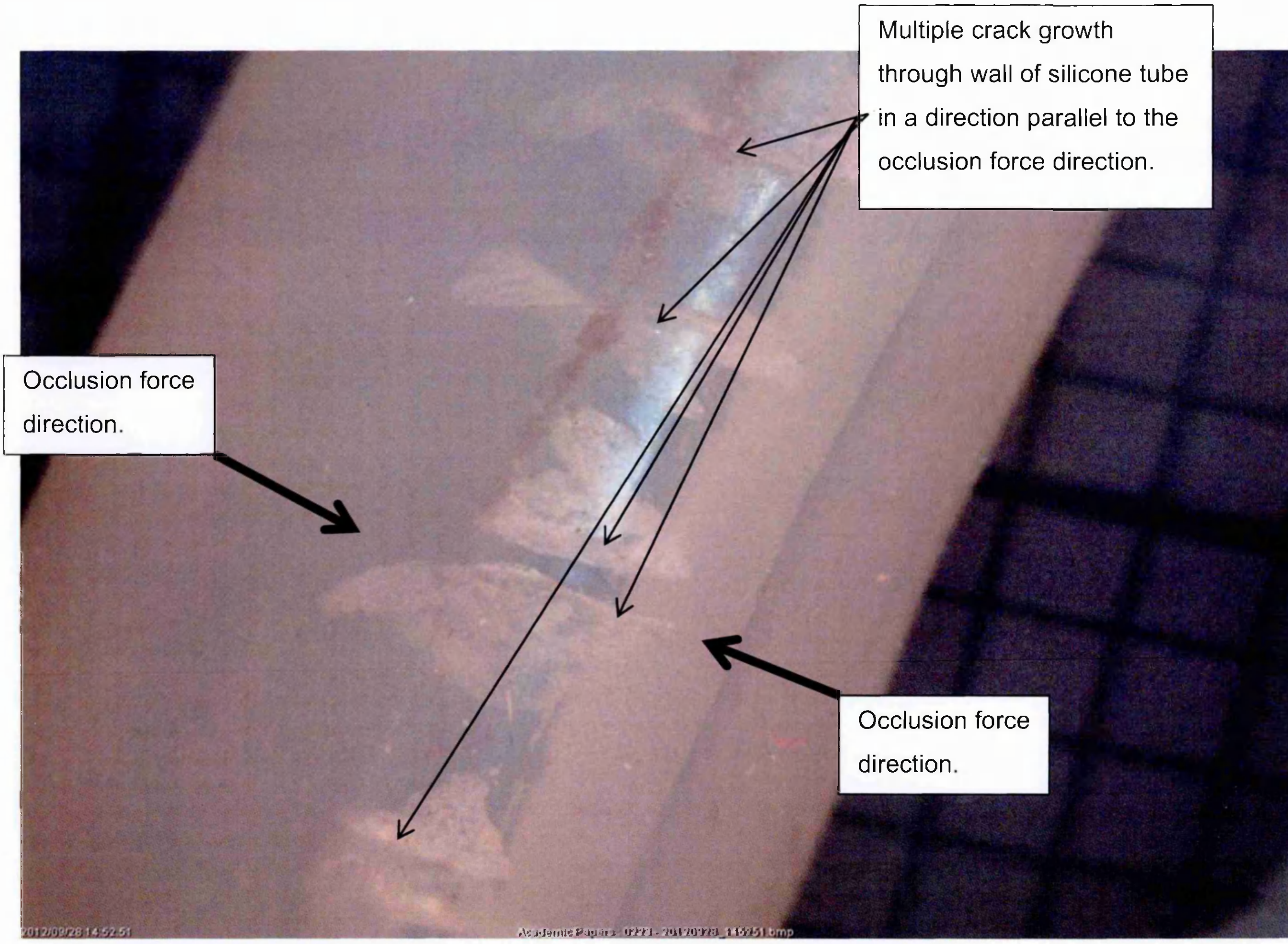


Figure 21 - Silicone tubing showing multiple crack growth in tube wall

Even with early signs of wear appearing the user is unable to realistically use optical checks of the tubes as an indication of life, due to the tube being held within a closed pump head and pumping fluid.

It is important to differentiate between early material changes, when the tube integrity is still intact and therefore still serviceable, and final tube failure: which is the occurrence of crack propagation within the tube wall to the point where a fracture path between the inner and outer bore occurs. If the tube fails in this catastrophic manner, where there is a fracture through the tube wall, it will spill the medium it is pumping.

For some users this may mean wasted time to clean up the spillage, which although inconvenient, can be built into downtime. However, for others this can be much more serious. For example, if a tube fails when delivering sodium hypochlorite, such as seen in figure 1, at a remote water treatment site – a situation still prevalent in many developing countries such as Brazil – and there is no backup pump present, the water being treated may not meet the required level of cleanliness, a result that not only impacts the water treatment company but also those customers it serves. For pharmaceutical companies who are using a pump to maintain the pH balance of a bacterial culture, a tube failure can mean the culture having to be thrown away and then completely regrown, wasting long periods of time and huge sums of money.

For all these users the tube itself is effectively the pump. With such widespread and growing usage of these pump types, being able to model the life of a tube and therefore predict when final tube failure is about to occur would allow a huge reduction of waste, whether it is time, resource or product. In this time of increasing strain on global resources, reduction of waste in any area has distinct benefits, both ecologically and commercially.

1.4 Life Modelling and Prediction

Predicting and modelling reliability and failure in products and systems has been widespread for a number of years and is the subject of numerous studies. A number of different approaches have been developed.

The use of failure mode effect analysis, FMEA, and root cause analysis, RCA, are among the tools that have been used to model and analyse system failure behaviour and they have been shown to help in the understanding of failure behaviour (35).

Condition monitoring has been shown in numerous studies to be a way of detecting and monitoring the degradation of products and systems (36) (37) (38) (39) (40).

Some studies have also built on this area to allow condition monitoring systems to be designed which incorporate an amount of intelligence. For example it has been shown by some (41) that the use of artificial intelligence knowledge-based systems, which use the experience of relevant experts on the system, can be used to facilitate fault diagnostics and improvement monitoring. It is here that we begin to see 'knowledge' being described as more than just data or information, but consisting of concepts, objects, relationships and inference rules. Key aspects of such a knowledge based system are:

1. An understanding of how the system it is being applied to works and the relationship it has with the outside world;
2. An intuitive understanding of how the system will behave when a certain subsystem fails;
3. An understanding of the 'symptoms' of failed subsystems.

These can be applied to the pump system and the tube within it; indeed within this study it is acknowledged that this approach could be used to not only help diagnose problems, but also predict them.

This idea is taken further by others (42), where artificial neural networks (ANN) are used to evaluate the expected failure rate of centrifugal pumps with some success. However this study also highlights that this method requires a large amount of data to facilitate a failure characterisation program, an issue noted by others (43), who acknowledge that predictive strategies need much more information than condition based strategies, including identification of abnormalities (deviations from normal behaviour) and prediction of residual life.

The need for large amounts of data is seen in the work of others, where in order to evaluate the on-going condition of a machine or system, some have used parameter monitoring applications through the monitoring of multiple signals. The signals chosen give an indicative level of health: for example, to look at tool condition in a machining process, force, vibration, acoustic emissions, temperature and power are monitored (44). In most cases the signals being monitored are 'indirect' signals, meaning that they can be monitored without affecting the system. When looking at peristaltic pump tubes a number of signals could be utilised to look at tube health, such as: flow, system pressure, temperature and speed.

Some have tried to simplify the characterisation of failure by utilising a pre-failure mechanism: Hannah (45) used a modified tube design in a patented concept dealing with tube failure. Utilising a tube structure with an inner and outer tube section, a failure of the inner tube would lead to leakage of fluid into an internal section which lies between the inner and outer tube, which is detected and allows the pump to be shutdown. However, it should be noted that this methodology requires a change to the tube structure itself making it inapplicable for existing elastomeric tubes.

The use of parameter monitoring, along with the characterisation idea seen in the knowledge based and artificial intelligence based system, has been seen to show the greatest potential to allow life modelling and hence prediction of peristaltic tube failure. This was seen in a reinforced tube (46), where through the monitoring of a single parameter, namely discharge pressure, some success was demonstrated. The model was built from empirical work on pressure monitoring, allowing a distinguishable pattern of behaviour to be observed for normal operation and pre-failure. There were severe limitations in this method, as it would only work in high pressure systems. However, it did show that precursors to tube failure could be recognised through a monitoring system.

The use of multiple parameter monitoring offers a robust approach, being able to gather the quantity and quality of data required to build a predictive system that can recognise what is normal behaviour and what signifies abnormal behaviour. To do this, the performance characteristics of a system need to be examined in detail and the most indicative parameters of condition or health chosen, utilising those ideas seen in the knowledge based system whilst trying to overcome some of the issues faced by the development of the artificial neural network system modelling.

It is important to note that a system which uses multiple parameters that works well for one method might not be the appropriate choice for the other. Hence, diagnosing mechanisms depending on a single sensor may not be able to make reliable results for the condition of a system, a point acknowledged in some studies (47) (48), or may limit the use of the mechanism as seen in the reinforced tube monitoring system described above.

It is also important that the tube monitoring and prediction employs the most effective analytical approach to the data used in the model. Studies have shown that pattern recognition approaches can be successful in developing prediction (49). Pattern recognition is based on a system showing a distinct behavioural pattern when failure is about to occur. One study (50) showed that similarity based modelling could be successfully used to develop prediction. This method is a non-parametric empirical modelling technique that uses pattern recognition from historical data to generate estimates of the current values of each variable in a set of modelled data sources. Similarity based modelling is looking to generate a predictive model based on the similarities between historically how a system behaved and how the system will behave in the future, one study (50) states that successful similarity based modelling requires:

1. Good historic data truly representing the equipment free from fault;
2. Sufficient historic data to properly encompass the patterns of interactions among the variables.

The coupling of a knowledge based system with a qualitative model based framework can compensate for insufficient data in the algorithm development, a fact acknowledged in a previous study (51).

Using a method based on a multiple parameter monitoring approach, coupled with pattern recognition and a knowledge based algorithm also allows the development of predictive algorithm to occur in stages, beginning with an analysis of the pump system to evaluate normal behaviour and the parameters which are most indicative of health. Building failure information onto this then allows iterative models to be trialled. This approach allows predictive maintenance and then expansion, to give greater and greater accuracy of the prediction model, allowing the pump users to achieve step by step improvements to their process, with the ultimate aim of utilising the full life potential of a tube, regardless of the environment it is working within. This systematic approach to design of a condition monitoring system has been used by others to cut down development times (52).

A conceptual algorithm design is shown in Figure 22. It allows for the use of previous knowledge in the form of empirical data and monitoring multiple parameters from the pump through sensors. These feed into an analysis engine which uses both the empirical data and the inputs from the pump to look for patterns in behaviour to predict how long a tube is likely to last and when tube failure is likely to occur:

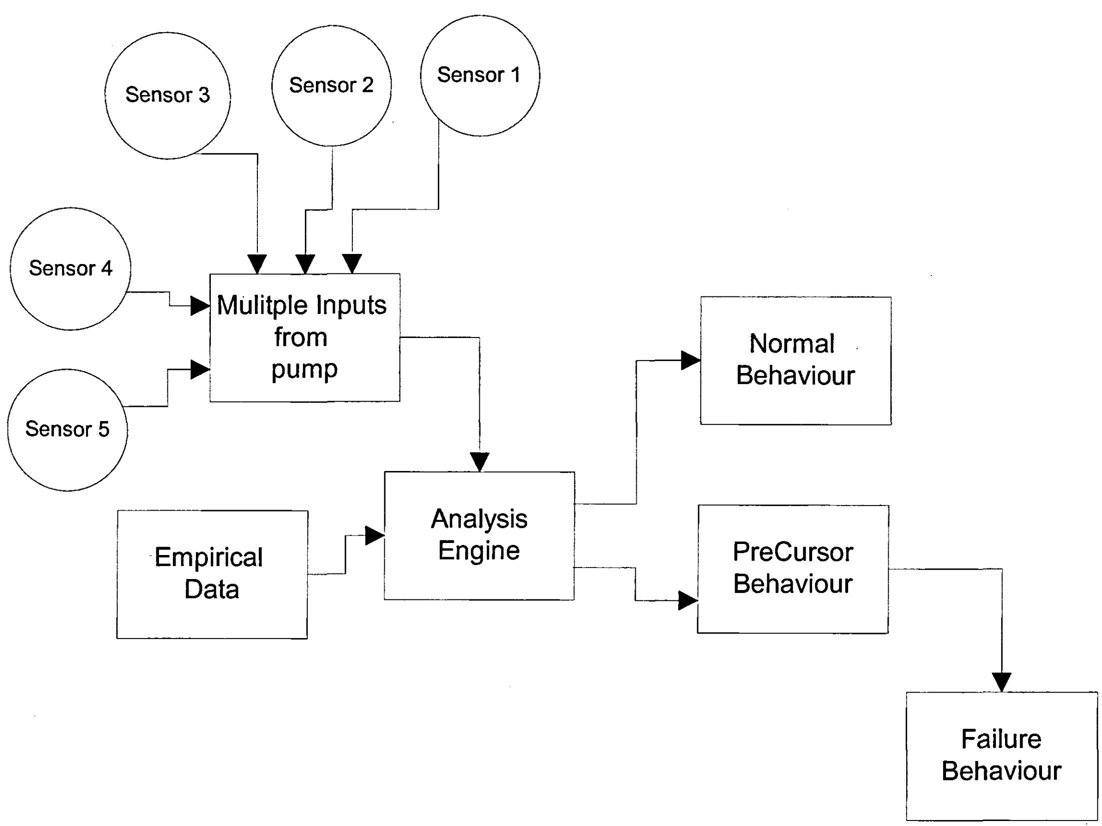


Figure 22 - Model of conceptual algorithm design

Modelling life and predicting when a tube in a peristaltic pump will fail relies on understanding the factors that will affect the life of a tube. To gain this understanding, both the life cycle of the tube and the peristaltic pump itself must be understood.

1.5 Scope of Study

The purpose of this research is to use the detailing of a tube life cycle from raw material through to failure within the pump as the basis to understand health for a peristaltic pump. This will allow the detail of the empirical model, shown in Figure 22, to be developed with clear relationships to an analysis engine. Multiple inputs from the pump to feed the analysis engine will be suggested, based upon the work undertaken. How normal behaviour could be defined will be outlined and precursors to failure behaviour will be put forward.

The author works as a design manager for a major peristaltic pump manufacturer and as such has utilised industry based information, the pumps made by the manufacturer and derived many test results from the company's knowledge databases. The manufacturer is interested in the results of this study and the findings to strengthen the position of peristaltic pumps within the pump industry by solving a number of problems faced by pump users.

1.5.1 Pumps in this Study

The peristaltic pumps used in this study come in a variety of sizes and designs to allow them to deliver a variety of flow rates, pressure capabilities, duty cycles and chemical compatibilities required for specific user environments. Within this body of work six key pump configurations have been considered. These allow a range of flow rates and thus tube sizes to be covered within the data analysis. It also allows key aspects of the pump head design to be understood, in terms of their effect on tube performance.

The functionality of the six pumps referenced is tabulated in the table below:

Unique Pump ID No	Track material	Roller or Shoe	No Of rollers	Flow Range	Roller material	Pressure Capability	Tube Clamping
1 – AW3	Glass filled ABS	Sprung Roller	3	0.45-1100 ml/min	Engineering plastic	0 – 2 bar	Continuous Tube
2 – BW5	Glass filled PPS	Sprung Roller	2	0.004-3500 ml/min	Engineering plastic	0 – 7 bar	Continuous Tube
3 – CWN	Glass filled PPS	Fixed Shoe	2	0.1-500 ml/min	Engineering plastic	0 – 10 bar	Fixed length of tube
4 – DFF	Glass filled Nylon	Sprung Roller	8	0.2-250 ml/min	Engineering plastic	0 – 2 bar	Continuous Tube
5 – EW6	Glass filled	Sprung	2	0.001-12	Engineering	0 – 4 bar	Continuous

	PPS	Roller		l/min	plastic		Tube
6 – FW7	Painted Aluminium Casting	Sprung Roller	2	1.16-2000 l/min	Engineering plastic	0 – 4 bar	Fixed length of tube

Table 2 - Pumps used in this study

All these pump-heads use a fixed track, but a couple of track materials are shown, both engineering plastics, such as glass filled polyphenylene sulfide (PPS), and painted aluminium. Rotor configurations concentrate on sprung roller designs, but a fixed shoe design is considered for completeness. Tube clamp arrangements and pump head arrangements that utilise both continuous tube and fixed length element design are considered

The scope of pumps has been chosen to allow the analysis engine to be based on as wide a range of empirical data as possible, thus ensuring that as many end-users can utilise the work as possible. The pumps chosen are manufactured by a pump manufacturer (the author's employer) with over 50% of all peristaltic pump sales world-wide, but it is envisaged that the principles applied are relevant to the majority of peristaltic pump designs, if not all.

1.5.2 Tubes used in this study

The tubes considered within this work are chosen so as to represent the needs of the major sectors of pump users, which can be split into industrial applications and sanitary applications. Sanitary applications cover the pharmaceutical, biotechnology and food processing industries. Here end-users are looking for tube materials which display low levels of leachables and food and drug contact approvals, such as given by the US government agency the FDA, Food and Drug Administration. The industrial applications such as waste water treatment, mining and chemical industry are looking for chemical compatibility, ability to cope with slurries, higher system pressures and often long life.

For the sanitary applications a range of hot vulcanised solid silicone rubbers are considered, using different raw material suppliers and a number of slightly different extrusion processes to produce the tube. Injection moulding of a hot vulcanised liquid silicone rubber is considered. The differences between the materials and the two processes are discussed, but the majority of the work concentrates on the extrusion production method and the tube thereby produced.

For the industrial applications a TPE, thermo-plastic elastomer is chosen with a broad chemical compatibility: Ethylene Propylene Diene Monomer (EPDM) / Polypropylene (PP) blend. This material is used in the extrusion of tube and it is this extrusion process that is considered in some detail later in the study.

These two materials allow an algorithm to cover a wide range of the end uses in the peristaltic pump market. Other tube materials are outside this body of work, but reference is made to key points of understanding that are applicable to these materials within the creation of a prediction mechanism.

2 METHODOLOGY

2.1 Overview

The understanding of tube failure within a peristaltic pump begins with the tube life cycle. From the raw material to the manufacturing process used to produce the tube; the peristaltic pump it is used within; to the working environment it is subjected to; and finally to the behaviour the tube displays up to the failure mechanism itself.

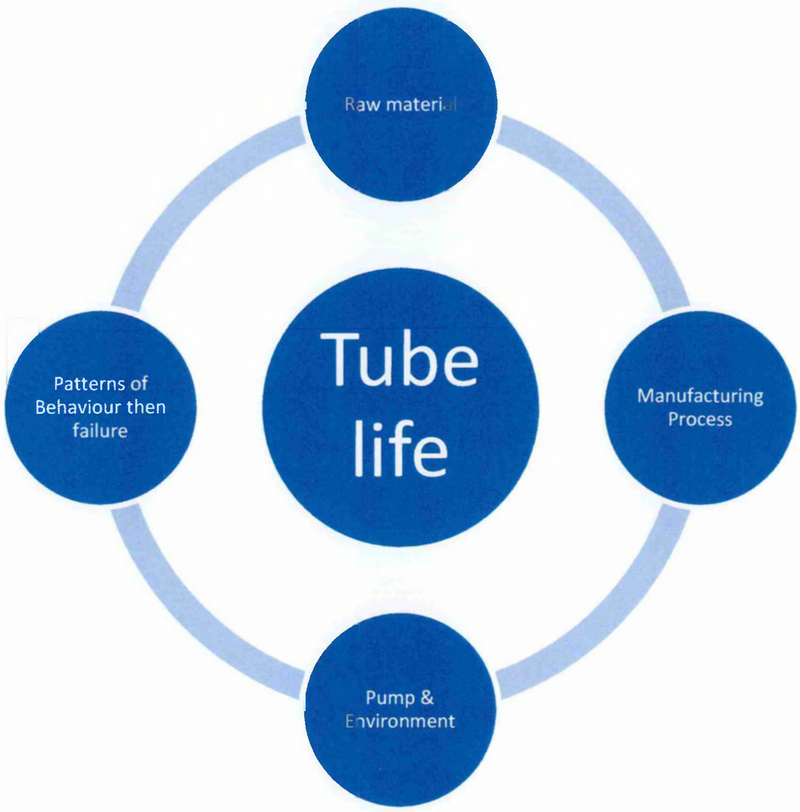


Figure 23 - Tube Life Cycle

By focussing on each section of this life cycle a comprehensive understanding of their influence on tube life can be built. This study takes the initial information available in each area and uses it to identify the knowledge gaps that then need to be filled; it suggests experimental routes that can be used to not only fill these gaps but to allow patterns of tube behaviour during its life to be established. Through these patterns, linkages between the different parts of the life cycle can be developed and additional experiments used to validate behaviour further. Within the pump and environment section, parameter monitoring is developed through the use of automated test equipment and indicative parameters for use in an algorithm are identified.

This study uses a systematic, almost prescriptive approach to build the information in each section, so that this methodology could be subsequently applied to different tube materials or manufacturing processes with relative ease.

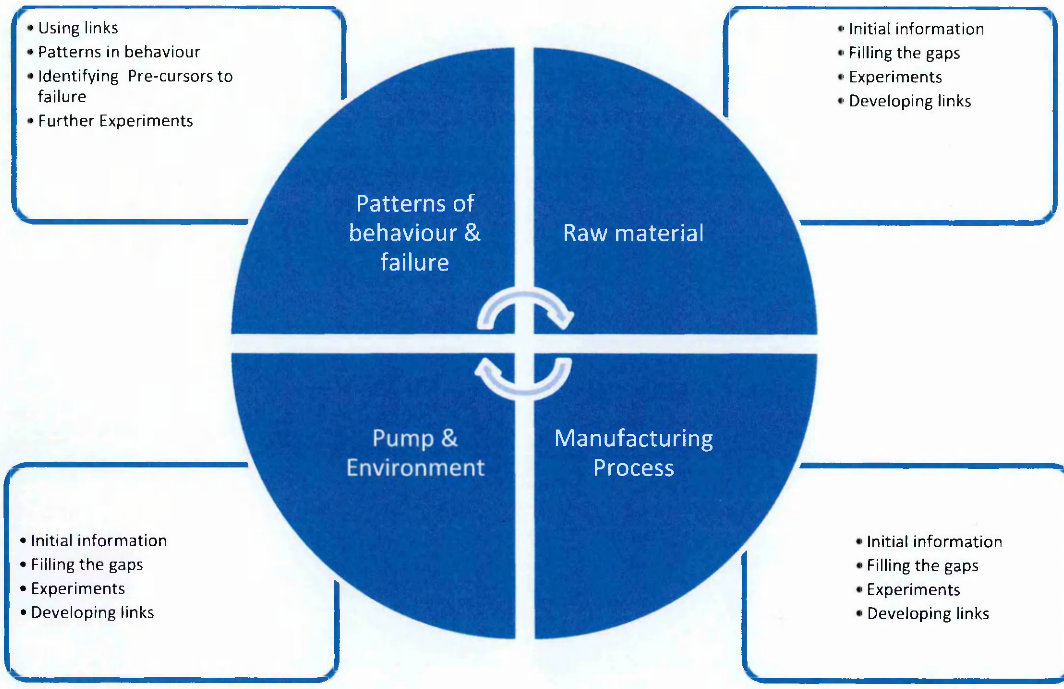


Figure 24 - Graphical representation of methodology

2.2 Sources of initial information

2.2.1 Confidentiality and IP

Intellectual property (IP) covers a wide range of information, such as patents, design and copyright. However, within any industry it is important to recognise that certain types of intellectual property, such as confidential information and knowledge formed and held specifically by a commercial organisation, can prevent the openness of communication that could make the filling of gaps in knowledge a quick and relatively easy process. This study has found that both confidentiality and intellectual property, as defined above, can make gathering data at certain stages in the development of a life cycle based algorithm extremely difficult and laborious.

Care must be taken when this is the case, as it may mean gaps in data and reliance on product literature for data. When this is the case commercial wording needs to be separated from factual data to ensure that an algorithm is based only on facts. It may also define the need for a greater number of experiments to confirm findings and ensure that the understanding is complete.

2.2.2 Raw material data

For most raw materials supplied to produce the tube the raw material data is presented from suppliers in a general format called a certificate of analysis. This certificate confirms that the material received meets the material characteristics outlined in a generic datasheet to a set tolerance. So for example a silicone rubber raw material may show results for the following:

- Material compression set
- Ultimate tensile strength
- Elongation at break
- Modulus at 250% elongation
- Tear strength

The certificate results are based on the testing of a compression moulded standardised dog-bone test plaque. The tests themselves are governed by either ISO or ASTM standards, depending on the raw material supplier's global position. Sometimes a company may use their own versions of these tests. This data has some limitations; it often is overly optimistic having been chosen from best results rather than all results and it is results from tests on compression moulded plaques. This is discussed further in later chapters, see Chapter 3.

Samples of certificates of analysis for the tube materials used in this study are included in Appendix 1: Certificate of Analysis for Silicone rubber and Appendix 2: Certificate of Analysis for EPDM/PP blend. Additional data such as polymerisation details, molecular weight of the base polymer, filler surface treatment, filler structural details etc. is more difficult to attain since it considered confidential to the supplier and therefore is not available to the author of this study. The methodology within this study therefore does not rely on this level of detail.

2.2.3 Manufacturing process data

Data on the manufacturing processes used in this study centre on extrusion lines. Each extrusion line utilised can be broken-down into its key substituent parts. By breaking the lines down in this way it is possible to evaluate the influences of each sub-section on the tube produced, in generalised terms without becoming so detailed that the learning points could not be applied to another material for which a prediction algorithm needs to be developed.

Generic extrusion conditions are often recommended by the raw material supplier, however this may need to be modified quite considerably to suit the specific extrusion line characteristics or the performance required of the end product, for this study that being tubing for peristaltic pumps. Data for each extrusion run is therefore stored on a standard recording sheet, called a run sheet, which allow any changes in key variables to be recorded so that they can subsequently be analysed as to their effect on the performance of the tube, once tube testing has been complete. Some data on the manufacturing process is considered the IP of the organisation which carried out the extrusion, the author's employer, therefore only broad methodology is discussed and not the detailed variables of the extrusion process, such as the specific mixers used the detailed temperatures and pressures in the extruder area. Run sheet examples are provided in the Appendix 3: Example of a production run sheet for extrusion of silicone rubber and Appendix 4: Example of production run sheet for extrusion of EPDM/PP blends.

2.2.4 Peristaltic pump and environmental data

Using the data set out in Table 2 - Pumps used in this study as a starting point, any experiments that utilise a peristaltic pump should record the following:

- Pump and Head type
- Number of rollers or shoes (to calculate number of occlusions of the tube)
- Speed of the pump
- Direction of rotation
- Ambient temperature
- Actual flow rate
- Change in flow over time (flow drop)
- Discharge pressure of pump medium
- Tube material
- Tube size
- Phase current (where applicable)
- Any database reference (if stored on a database)
- Unique ID to allow cross-referral
- Pump medium details

These details are recorded in a database or a spread-sheet as appropriate, a sample of which is shown in the Appendix 5: Data entry sheet for database logging of experiments.

2.2.5 Exploring data already held

There are a number of different ways that data can be analysed, the aim of the first stage of analysis is to 'get a feel' for the data, to look for patterns of behaviour: through analysis of graphical representations in a number of

forms or the use of a variety of distribution models; using statistical tools; or simplifying data to look at one or two aspects such as the mean or standard deviations.
This study uses a variety of methods to build up an understanding of the 'system' and its relationship with the outside world: the system being defined as the tube within the pump. It is looking at patterns that can be used to demonstrate health and indicators thereof. It is also looking for patterns or incidents that can act as pre-cursors or symptoms of failure.

It is acknowledged that in order to build a predictive algorithm for the life of a tube it is important that enough information is gathered on the performance of tubes themselves, both in terms of actual life and the way they change when subjected to different pumping environments, such at different system pressures. A point acknowledged earlier in the study following a review of the work of others. This study therefore has utilised the historical data on performance from the database of the author's employers. However this data has to be viewed with some care since the experimental set up and results are open to both experimental error and interpretation respectively. However more rigorous systems have been developed over the period of this study so it is suggested that the level of error has been reduced although this needs further quantification.

2.3 Experimental set-up

A variety of experiments are required to fill the gaps in knowledge needed to complete a life cycle analysis and create an algorithm that allows life prediction. Experiments to look at how the pump environment influences the life of a tube will differ to those that look at the raw material characteristics that influence tube performance. Experiments to look at the extrusion process and how it can be changed to affect the performance of the tube produced will differ from experiments which focus on a specific characterisation of the tube as it changes over time. However, within each experimental set there needs to be the development of a method which allows the recommendation of a standardised methodology for further work in the development of the predictive mechanism.
There are five key areas of experiments where standard methods have been developed:

a. **Raw material** – identifying key indicators to use to find the characteristics that most influence tube performance

b. **Raw material & Process** – using extruded dog bones and key mechanical tests to analyse the raw material, bringing the same process used to produce the tube to the production of test samples.
c. **Process** – breaking the extrusion process into stages to understand key influences on the tubes produced, whilst identifying where work can be done to narrow the performance distribution of tube produced to aid tube life estimation.
d. **Environment and Pump** – using DOE and Taguchi analysis to explore those factors that influence tube performance, including life and pre-cursors to failure, along with standardised test stations to remove 'noise' for an experiment and 24 hour monitoring to capture live data when appropriate.
e. **Tube Life Cycle** – developing a holistic approach to the tube, developing key linkages between each stage of the life of a tube to allow interactions to be understood more fully and monitoring parameters to be developed which can expose health.

2.3.1 Raw material key indicators - methods to analyse

Raw materials used to produce peristaltic tubes can initially be characterised by using the material information provided by the raw material supplier. However, this information can be expanded further through some key tests depending on the material type, using techniques that can be completed in a simple laboratory environment or are readily available throughout the analytical industry.

For silicone rubber the following tests can be utilised:

1. Total extractables test, as set out in the FDA standard 21CFR 177.2600 – to quantify the level of extractable, un-cross-linked material, present in the material studied.
2. Comparative swell tests in toluene – to study the level of cross-linking in comparable materials.
3. Headspace Gas Chromatography/Mass Spectroscopy (Headspace-GC) - to look at key volatiles extracted from the materials studied
4. Deformation changes over time, using flow rate drop indictors – to study how different but comparable materials deform.

For EPDM/PP based materials the following tests can be utilised:

1. Fourier transform infrared spectroscopy (FTIR) – to study the different compounds present in comparable materials to see how they differ.

2. Comparative thermo-gravimetric infrared spectroscopy (TGIR) – to study the different compounds present in comparable materials to see how they differ.
3. Differential Scanning Calorimetry (DSC) – to study the comparative polymer mobility in comparable materials.
4. Nuclear magnetic resonance (NMR) measurement of swell tests in toluene after extraction via xylene – to quantify the level of cross-linking within the EPDM phase of a Santoprene blend.
5. Transmission electron microscope (TEM) – to allow observation of microstructure of a Santoprene blend.
6. Comparative swell tests in toluene - to quantify the level of cross-linking within the EPDM phase of a Santoprene blend using a simply methodology.

It has been found that carrying out these tests using the tube in its final form allows links to be drawn between the raw material, manufacturing process and pump environment much more quickly. In fact a number of industry studies have shown that trying to characterise the key material indicators for good pump performance though pure compression moulded plaques proves to be unsuccessful (10) (11). The techniques used in this study do not preclude the use of other techniques that may be available, such as scanning electron microscopy, (SEM) or optical microscopy, they are however the techniques that the author has used to highlight key material characteristics important to life cycle analysis.

2.3.2 Using Dog-bone tests

The tube geometry as it is fabricated can prove difficult to extract samples for some test processes. In these cases it has been found that using dog-bones produced in the standard compression moulded method can offer a broadly comparable set of results. However these results can be made more relevant by using the same process to produce the dog bones as the tubing; for extruded tubing this is by extruding a flat sheet then punching out a standardised dog bone to carry out the material tests. This is shown for an EPDM/PP blend, Appendix 6: EPDM/PP Dog bone vs. tubing test results. The difference between results using compression moulded plaques and the results of the same test using an extruded plaque can vary considerably. For the EPDM/PP blend tested a certificate value produced from a compression moulded plaque shows a modulus value of 6.1MPa @ 100% strain, whereas extruded plaques of the same material show this modulus is reached at between 25% and 40% strain values.

Comparing the mechanical properties of this extruded plaque to that of the tube shows a close linkage in properties, this can then be used to complete a life cycle analysis. This linkage is discussed in more detail in later chapters. The study found that the complexity of the silicone rubber extrusion and crosslinking process did not allow an effective extruded plaque to be developed within the scope of the work; however other process experiments can be used to explore the links between tube and process more readily.

Establishing this link early in a life cycle analysis begins to close the gaps between each stage of the life cycle, bringing raw material and end tube performance closer together.

2.3.3 The use of Design of Experiments (DOE)

Design of experiments is a methodology for systematically applying statistics to experimentation. It is defined more precisely as a series of tests in which purposeful changes are made to the input variables of a process or system so that one may observe and identify the reasons for these changes in the output response (53).

By carefully designing an experiment the influence of independent variables on the response can be ascertained, as can interactions between variables. This study uses a two stage strategy to the experiment design:

1. Limited Response Surface Stage: this has as its basis a small number of possible factors, which results in key factors being identified, but gives limited information on interaction.
2. A full Response Surface Stage: Where the use of a few key factors, identified in stage 1, can then are used to look at interactions.

The study looks to identify the key two to six factors, the interactions between them and wants to predict outputs, (responses), from inputs, (variables). The need for multiple responses requires trade-offs;

- The optimum may be outside the operating range of the experiment
- Experimentation is limited

However this study is looking for consistency of responses rather than optimality.

Factors are classified as;

1. Control Factors: those that can be set at a specific level, e.g. temperature, pressure, etc.
2. Noise Factors: those that could be controlled but at great expense, e.g. batch, environment etc.

Some control factors may be fixed due to circumstances, e.g. tube wall thickness.

The responses also need to be determined and how they will be measured considered carefully. The choice of experimental design, such as shown in Appendix 17, takes all these things into consideration.

This study uses both DOE and the more traditional approach of varying one factor at a time. Set within an industrial environment this study is subject to commercial restraints, namely financial limitations, which has limited the amount of complete randomisation when using DOE.

2.3.4 Process control and extrusion trials

Within the process for extruding tubing there are a number of key sections of the process where the modification of process parameters can influence the material properties. These will differ between silicone rubber extrusion and the extrusion of the EPDM/PP blends discussed in this study. In order to analyse the influence of the parameters in each key section and how they interact with each other as discussed above, a number of differing methods are used.

The Silicone extrusion process parameters explored are:

1. Mixing time –the mixing time can influence the distribution of catalyst, used to activate the cross-linking process, within the raw material it is being mixed into. It can also influence the distribution of filler within the silicone material. By altering this parameter the study is able to explore how this could influence silicone tube performance.

2. Thermal energy to which the material is exposed on the extrusion line – this parameter is explored to look at how the amount of heat that the material is exposed to influences the rate of cross-linking and the amount of cross-linking that occurs within a silicone material.

3. Extrusion line run speed – the speed at which the extrusion line is run affects the time the material is exposed to certain levels of heat. By adjusting this parameter the combinational effect of line speed and temperature on the final tube performance can be explored.

The EPDM/PP blend extrusion parameters explored are:

1. Extruder screw speed – the extruder speed can be adjusted to look at how the mixing within the extruder screw can affect how well the subsidiary materials within the Santoprene are both distributed and effected by the mixing action, by adjusting this parameter it is possible to look at the influence this has on the tube performance

2. Extruder temperature – the extruder temperature settings influence the level of mix within the extruder screw, by adjusting this parameter it is possible to look at the effectiveness of the melt mixing and the possible effects on tube performance.

3. Extrusion line run speed – the extruder line run speed helps govern the tube dimensions achieved by the extrusion line. By adjusting this parameter in combination with the screw speed and temperature the influence on tube performance can be observed.

2.3.5 Mechanical testing - compression testing and tensile testing

Tensile testing on extruder dog-bones and on tubing utilised the standard test equipment and methodology outlined in ISO37:2005 which describes a method for the determination of the tensile stress-strain properties of vulcanized and thermoplastic rubbers. The properties which can be determined are: tensile strength, elongation at break, stress at a given elongation, elongation at a given stress, and stress at yield and elongation at yield. The measurement of stress and strain at yield applies only to some thermoplastic rubbers and certain other compounds. A typical testing system would comprise a universal testing machine, with either mechanical self-tightening, or pneumatic grips, see Figure 25. An extensometer is usually required to measure specimen elongation, and a mechanical contacting extensometer or a non-contact video extensometer can be used to measure the separation of gauge marks applied to the specimen.

Compressions testing of samples used equipment unique to this study see Figure 26.

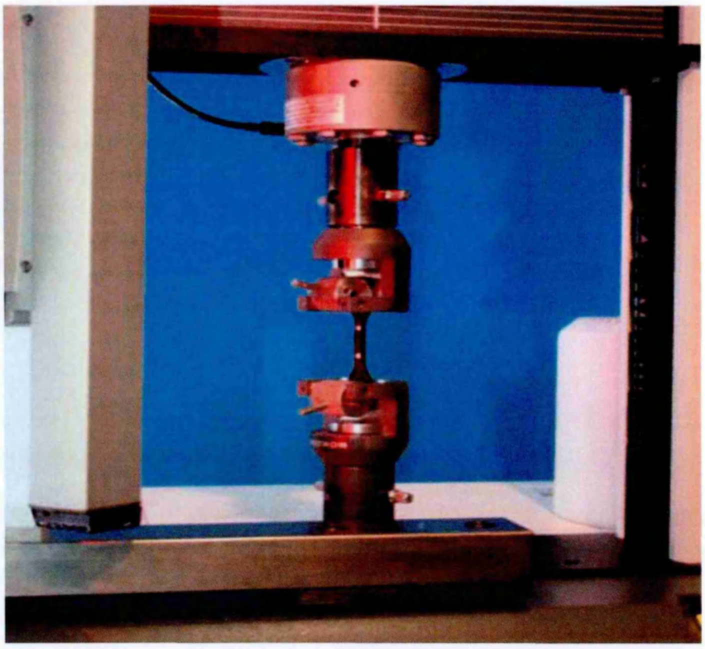


Figure 25 - Typical ISO37 Test equipment (Source www.instron.com application note)

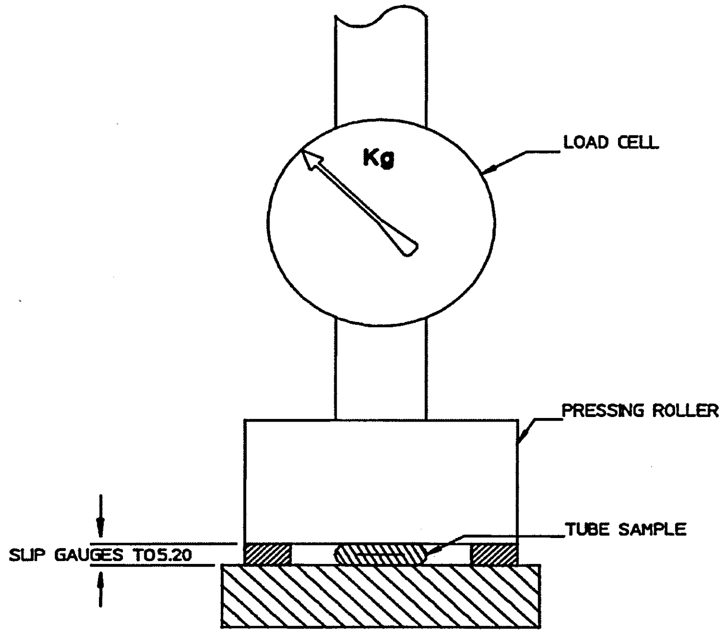


Figure 26 - Compression testing equipment

2.3.6 Experiments for gathering data on environmental factors

The methodology for gathering data on environmental factors that influence life, i.e. system pressure, temperature and pump speed, is based on the use of historical empirical data held by a peristaltic pump manufacturer, see section 2.2.5 'Exploring data already held' for caveats. The data is analysed to ascertain if key factors, such as temperature and pressure, can to be used in the construction of an initial DOE matrix: this is used to validate the data held and suggest further experimental sets that are required to fill gaps in the data. An understanding of environmental factors that are either material independent or material dependent can also be established. These experimental sets also bring to the fore the parameters that can be measured to ascertain the health of the system under the influence of environmental factors. It should be noted that the experiments within this study use water as the pump medium, due to health and safety restraints, however the importance of the effect of the pump medium on the performance of the tube is discussed in future sections see 5.3.1.6 and 5.3.2.4.

2.3.7 Standardising Test Stations

It is important when carrying out experiments to look at a set system that the system components are standardised as much as possible to avoid the influence of experimental noise. For that reason a number of experiments looking at environmental factors utilise a standard set-up. A photograph of standard set-ups used is included in the Appendix 7: Standard test station layout. Any test equipment used will be subjected to regular calibration, for example the flow meters used have an annual maintenance check by the an independent test house.

2.3.8 Using Automated Test equipment (ATE)

Automated test equipment or data acquisition equipment is widely used throughout a number of industries, including the one in which the author is employed. Within this study the use of ATE allows 24/7 measurement of flow and pressure. This continuous measurement allows the performance of pumps to be monitored in the detail required to build the empirical data needed for a predictive mechanism. However, there are some experimental sets where the ATE was unavailable for commercial or confidentiality sensitive reasons and where this is the case, manual flow and pressure measurements have been recorded along with the frequency of the measurements and any potential error that may have been encountered due to the measurement methodology or frequency of measurement. Any automated test equipment used will subjected to regular calibration, for example the pressure transducers used have an annual maintenance check by the an independent test house.

2.4 Data Collection

The gathering of data in all aspects of life cycle analysis is lengthy and as a result there are a number of points to make in this area.

2.4.1 Life and Performance of Tubes

Life in hours and in number of occlusions is recorded for all tubes, along with their flow drop characteristics. However, there are a number of results where some detail has to be omitted due to the sensitive nature of the data and the resultant IP issues associated with it. Where this has been required it will be noted clearly within the results. There are also results where a test is suspended before failure but the results are still used within the analysis, where this is the case it has been noted.

2.4.2 Observation of failures

The point has been made regarding the commonality of fracture position for the tubes utilised during this study. This was concluded following the observation of a large number of tube failures from both materials studied. A more detailed examination of a smaller sample of silicone tubing was also carried out as this material displayed a greater level of early material change, see Appendix 8: Observations of failure for silicone rubber tube. From this work a number of typical fractures and damage types are shown in section 3.2 to show how this data can be used to help in the identification of 'normal' behaviour. A number of 'abnormal' failure modes are also discussed in section 3.2, these show the need to continue with failure mode observations; this ensures that the algorithm that has been developed to monitor health is relevant to the system it is being used to monitor.

2.4.3 Date Accuracy; Incomplete or absent data

Where historical data has been used the level of confidence in some of the data may be low, this is due to experimental set-up being sub-standard, the frequency of result gathering being low or the analysis of results to being suspect. Where the level of confidence in the results is low or where experimental error is suspected some filtering of historical data will be carried out to remove suspect results.

Data gaps can occur due to confidentiality issues, in these cases some detail may be partially included to avoid disclosure issues whilst still offering meaningful results. Some experiments may have been stopped due to commercial reasons, in these cases suspensions may be utilised during statistical analysis.

2.5 Calculations

2.5.1 Swell methods

Rubber generally swells in organic liquids, swelling of a rubber compound by absorption of liquid results in a swollen elastomeric network. It is dependent on the solubility characteristics of the rubber and the fluid in which it is immersed. It can be used to ascertain the relative crosslink density in cross-linked rubbers such as PDMS and EPDM, and this has been studied in detail by others (54). The theory of rubber elasticity (54) outlines that a material will show rubber like properties if the following three requirements are met:

1. The presence of long-chain molecules, with free rotating links;
2. Weak secondary forces between the molecules;
3. An interlocking of the molecules at a few places along their length to form a three-dimensional network.

Within cross-linked rubbers the third requirement is satisfied by the introduction of cross-linkages between the polymer chains at various points along their length. The greater the level of cross linking the more closely bonded the network, hence a restriction in its ability to swell

Using the work by Treloar (54) a simplified swell method can be applied for comparative analysis. It is known that the higher the swell rate the lower the cross-link density, this is checked against the life performance of the tube and the amount of deformation that occurs subsequently in the tube that was subjected to the swell test. Initially a solvent with the correct solubility parameter must be chosen. The best swelling agents for a given polymer are those whose solubility parameter are nearest to that of the polymer, and which are therefore more closely related in chemical structure to the polymer (54). Using compiled data (55) for the PDMS silicones and EPDM rubber blends being used in this study, toluene is an appropriate choice with a solubility parameter of 8.97. The material undergoing the swell test and the solvent to which it is exposed must reach equilibrium, that is to say, when the transfer of liquid from the solvent into the material has stopped. The swell experiments are carried out at a normal room ambient of 23°C.

At this stage the swell equation can be completed:

$$\boldsymbol{SR} = \frac{\boldsymbol{Ws - Wo}}{\boldsymbol{Wo}} \qquad \text{(Equation 3)}$$

Definitions:

SR = Swell Ratio

W_s = Weight post swell test

W_o = Weight pre swell test

Extraction methods are used in more detailed swell testing experiments on the EDPM/PP blends to clarify whether the simplified method could also be used for them as a comparable measure. EPDM/PP blend samples are extracted with hot xylene to extract the polypropylene phase from the blend. The samples are then dried in a vacuum, before being swollen in toluene to give an indication of crosslink density. H-NMR, nuclear magnetic resonance spectroscopy technique which allows the determination of the molecular structure as well as the spatially resolved representation of the molecular mobility, was used with this extraction method.

The simplified methodology shown by equation 3 was checked through several measures:

- Samples were immersed over a period of time up to 96 hours to identify the point at which they no longer gained any further weight, thus signifying that equilibrium had been reached. It was found that 48hrs was sufficient time to reach equilibrium in the experiments carried out, Appendix 9: Swell Measurement.
- Multiple sample geometries were tried in each test using material taken from adjacent tube positions and tested. It was found that when carrying out comparable testing between two different tube materials, tubing of the same size should be used to cut samples with a weighed tolerance of +/- 10% to avoid results that are not comparable, Appendix 10: Swell Measurement – variation with geometry.

- Pre and post swell measurements were duplicated ten times to check the experimental variation that could occur in the weighing method employed, having an element of manual intervention. The minimum, maximum and average were plotted to look at the error level that could occur in measurement and subsequent swell calculations. It was found that the weighing method could introduce less than a 5% error, Appendix 11: Swell Measurement – weighing method accuracy.

2.5.2 Converting life into number of occlusions

Life in number of hours is recorded for each tube tested, however where comparing performance figures different patterns can become clear when the number of hours is converted into the number of occlusions the tube has been subjected to:

$$\mathbf{O_n = (rpm \times 60) \times R \times t} \qquad \text{(Equation 4)}$$

Definitions:

O_n = No of occlusions

R = no of rollers in head

t= life in hours

2.5.3 Thermal energy applied to silicone

During the extrusion process heat is applied to the silicone material at various stages: the extruder, in the hot box oven and within the line oven. These parts of the extruder line are detailed within section 4.2. The amount of thermal energy the material is exposed to during the process will influence the level of reaction that occurs. It is therefore necessary to capture this at least in a simplistic form to allow general comparisons between materials and extrusion line types. This is shown in detail in 4.2.4.

$$\mathbf{T_n = T_a \times t \times (\frac{V}{A})}$$

$$\mathbf{T_{total} = T_n + T(n + 1)} \qquad \text{(Equation 5)}$$

Definitions:

T_n = total energy for section n of the extrusion line

T_a = temperature applied at the section

t = time at the section

A = surface area of material exposed in that section

V = Volume of material exposed in that section

T_{total} = total energy applied over entire line

2.5.4 Relating flow drop to deformation

It is known from equation 2 that Q is directly related to pillow volume. It is also known that the pillow volume will change as the tube material changes. Permanent deformation of the material will result in permanent pillow volume changes. Therefore by looking at the change in Q over time we can see how the pillow volume has changed and therefore how much the material has deformed.

$$Qd = \left(1 - \frac{Qn}{Qo}\right)\% \qquad \text{(Equation 6)}$$

Definitions

Q_d = Flow drop after n hours as a percentage

Q_n = Flow at n hours

Q_o = Flow at start of test

2.6 Presentation of Results

The results of the life cycle analysis are shown for a tube in two distinct materials. The results show the raw material information from the supplier and process information from initial extrusion runs. The results of experiments that are done to build up the gaps from the analysis of this initial data are then shown. This is shown for a number of tube sizes and indicates where results suggest a characteristic which is size dependent and those which may be size independent. It will link key material characteristics to tube performance exposing those characteristics that are significant in acting as symptoms of 'health' and how they can be measured using parameters that can be monitored. It also highlights how the monitoring is carried out and where the use of sensors can be employed. The results also show how the tube and its materials are linked to the pump and the environment it is in.

The raw material information is presented as the original certificate value. This is then validated using results from key indicator tests. Key indicator test methods are noted and detailed where non-standard methods are employed.

Historical data is presented as normal distributions. Key factors which are first identified by the historical work are shown and the links to the experiments within this study are highlighted.

The experiments show the Taguchi matrices employed and the analysis of factors, showing those that are significant. The DOE results also show interactions between factors where they occur. The data from a single tube size and pump type is then analysed using a statistical tool developed by the author's employer to analyse to look at how suitable sample sizes from a population which give known reliability and confidence levels can be determined. This method is shown in Appendix 21.

Results of mechanical tests, both compression and tensile are shown in N/mm^2 format.

The experimental set-up for the gathering of this data if non-standard is highlighted and shown within the appendices. Results of extrusion trials in terms of the performance of the resultant tubing is recorded by using DOE factorial analysis, and by plotting flow drop off as an indicator of tube health. Where material analysis of the resultant tube has been carried out this is shown.

3 TUBING MATERIALS

3.1 Overview

The widespread use of silicone rubber and the EPDM/PP-blend-based materials by peristaltic pump users means they form an important part of any life modelling and predictive algorithm created. Looking at a range of silicone rubbers and EPDM / PP TPVs offered to the industry as suitable for the manufacture of peristaltic tubing allows this study to look at the complexities in the material that make a good peristaltic pump tube.

It is at this point that it should be quantified what is meant by a good peristaltic pump tube from a modelling and prediction point of view. Consistent performance in terms of life and flow performance is much easier to model and hence predict than tubing that fluctuates widely in these aspects. It is important to note that the stresses that occur in a peristaltic pump are complex. Recent work to model them has provided some information (11); this is discussed in more detail in chapter 5. However a number of key observations can be made on the tube failure modes and fractures.

3.2 Tubing failure modes and fractures in Peristaltic pumps

The development of a life modelling methodology should not have to rely on the detailed study of the fracture. The methodology is focussing on patterns of behaviour prior to final failure not the fracture post-failure, at this point any prediction is irrelevant. However, it is important that an end user can use the observation of fracture position to identify 'normal' behaviour with regard to fractured tubes.

The tube fracture site seen within the peristaltic pumps in this study shows consistency of position on the tube regardless of the pump head type used, see Figure 27 and Figure 28. Therefore a 'normal' or expected typical expected fracture position can be quantified. Fracture positions that fall outside this normal pattern can be used to highlight where further investigation and study may be required as the tube behaviour could be deviating from that being used to model life in this study.

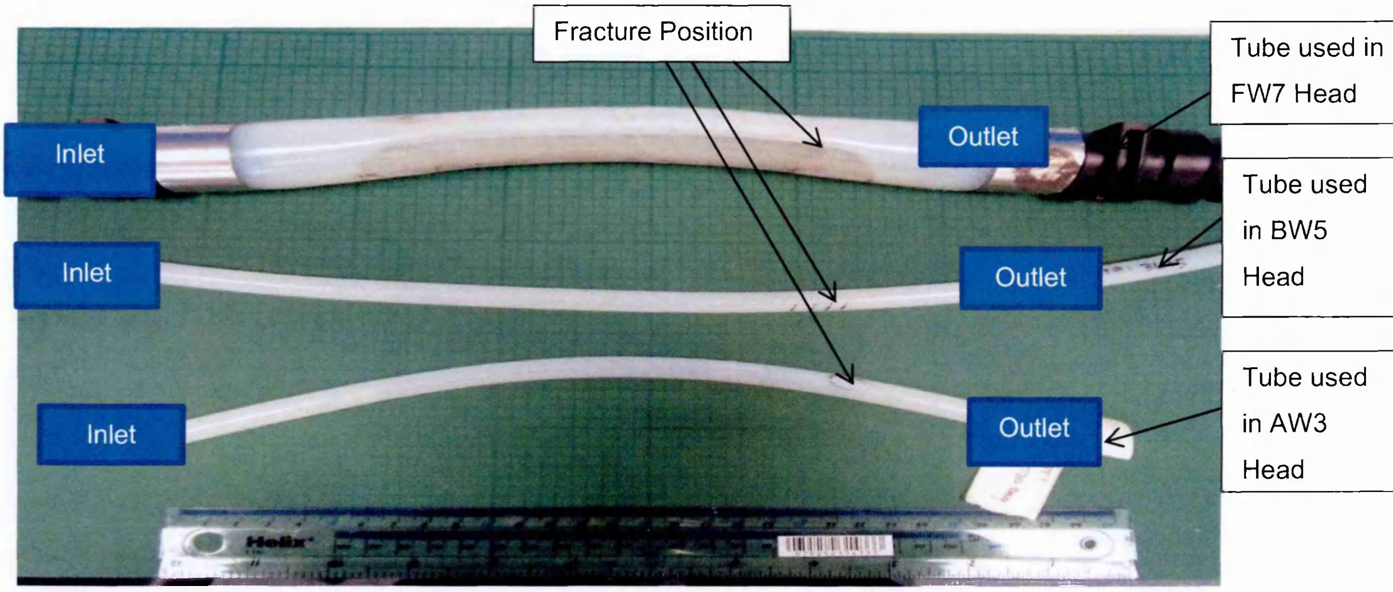


Figure 27 - Typical fracture position on silicone rubber tubes used in different pump types

Looking at the typical fracture position shown in Figure 27 and Figure 28 it can be seen that these match the high stress areas as discussed in section 1.3 and shown in Figure 11 and Figure 12, with the majority of the typical failures seen at the outlet end of the tube and in the high stress area. Failures can be seen on both the inner and outer wall as defined in Figure 13 on page 17.

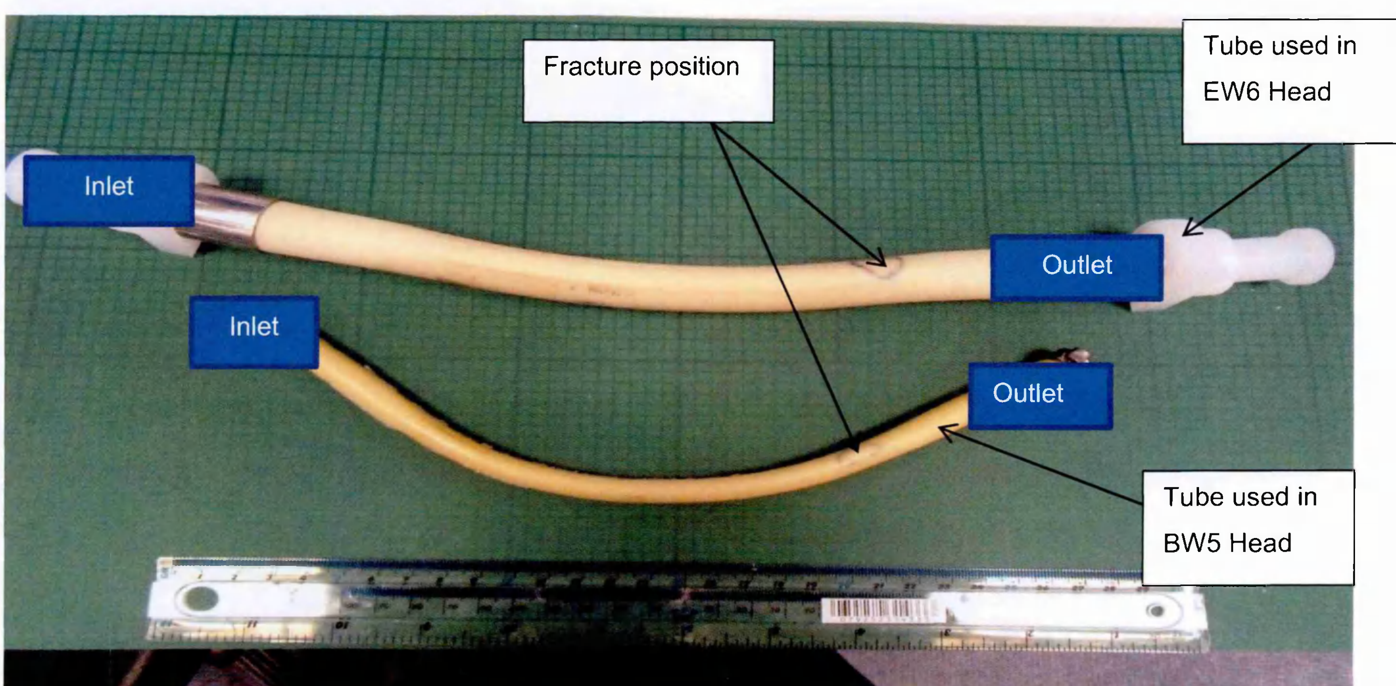


Figure 28 - Typical fracture position on EPDM/PP blend tubes from different pump types

The end user may also need an awareness of the sort of fracture which falls outside the general patterns discussed in section 1.3. This includes situations where a pump head is performing outside normal expectations, for example when the occlusion force is much greater than anticipated, or wear of parts causes uneven occlusion or environmental effects which have not been modelled as completely as required. These situations can result in mechanical wear on the tube, see Figure 29, Figure 30, Figure 31 and Figure 32, environmental stress cracking, see Figure 33 and Figure 34, combinational effects, see Figure 35 or increased shear damage on the tube surface see Figure 36.

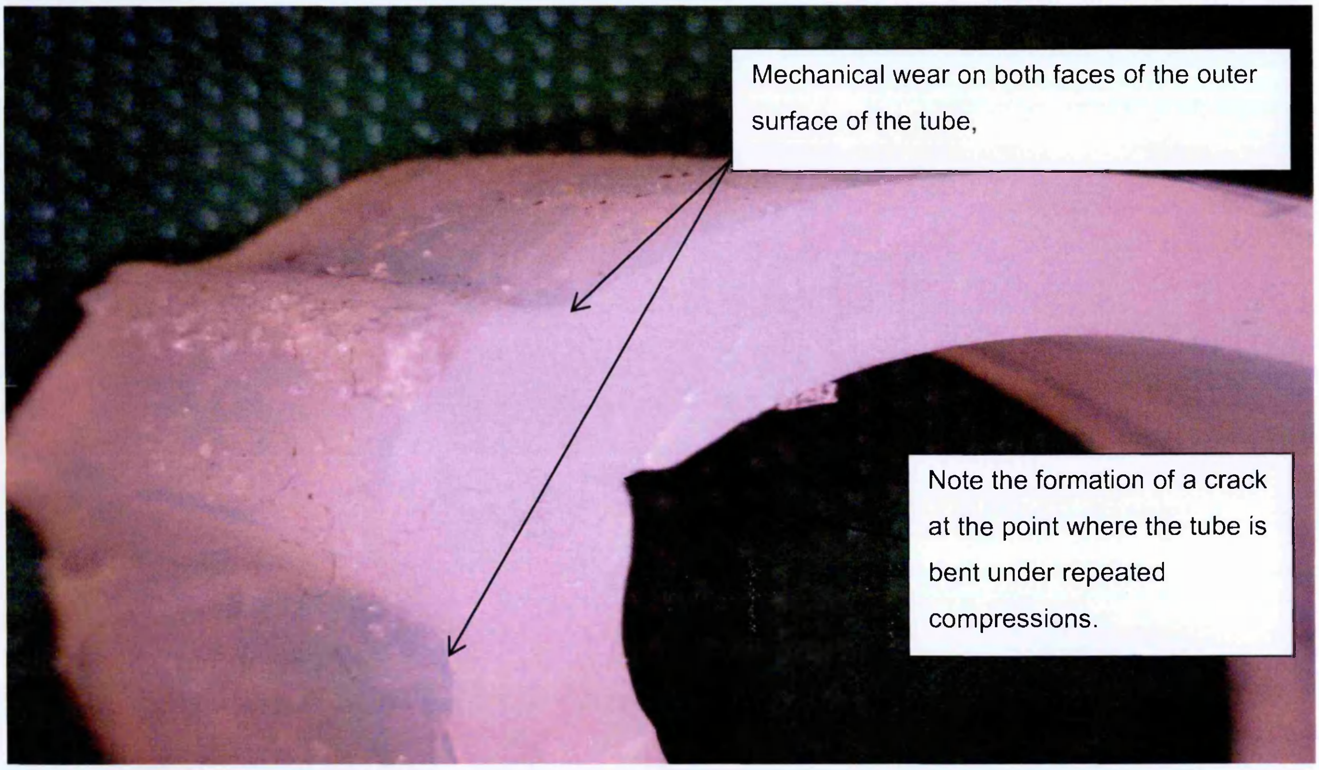


Figure 29 – Section 1 through a silicone rubber tube showing damage due to mechanical wear

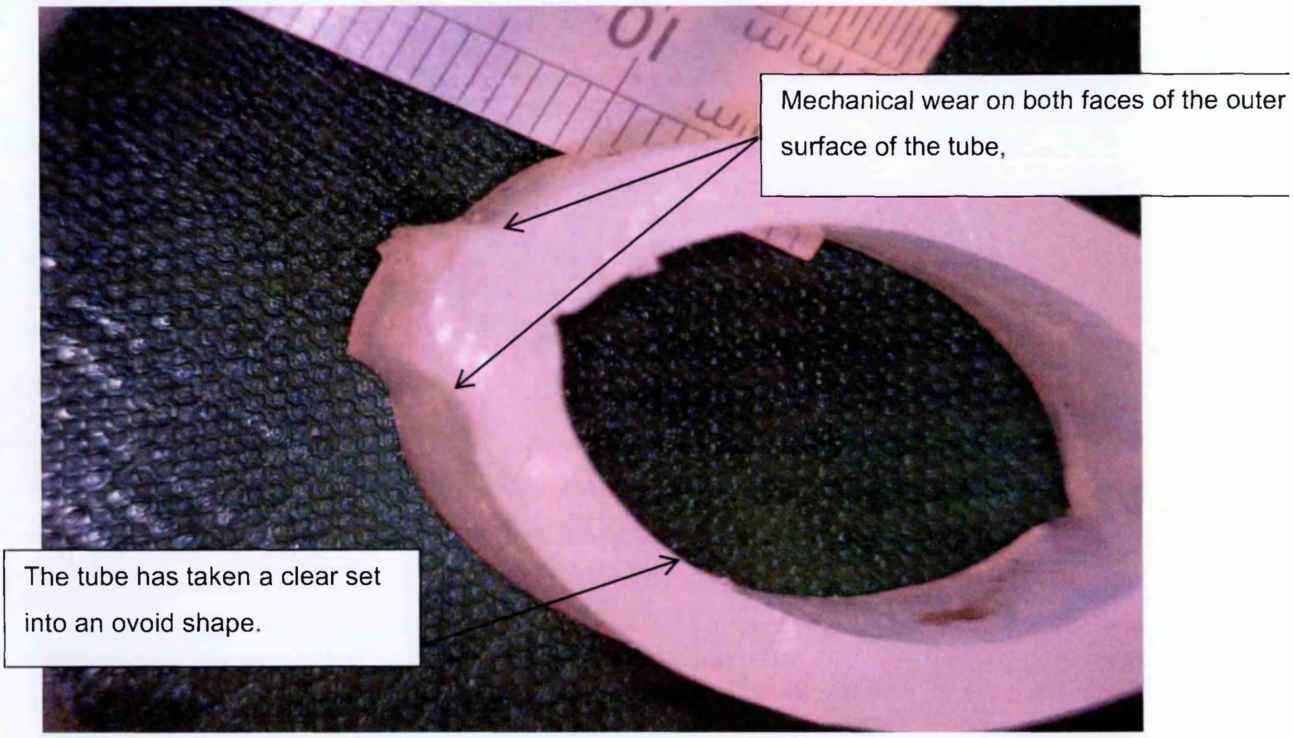


Figure 30 - Section 2 through a silicone rubber tube showing damage due to mechanical wear

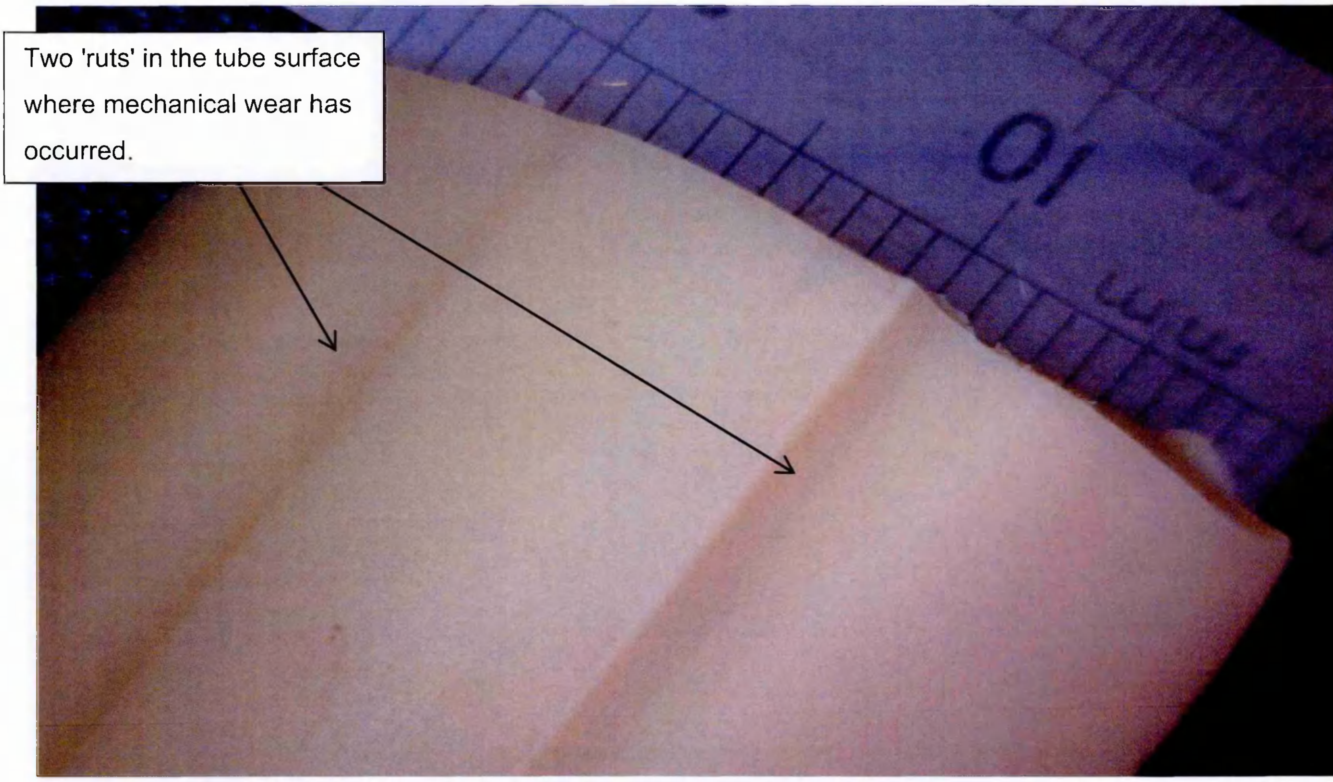


Figure 31 – Section 1 through an EPDM/PP blend tube showing damage due to mechanical wear

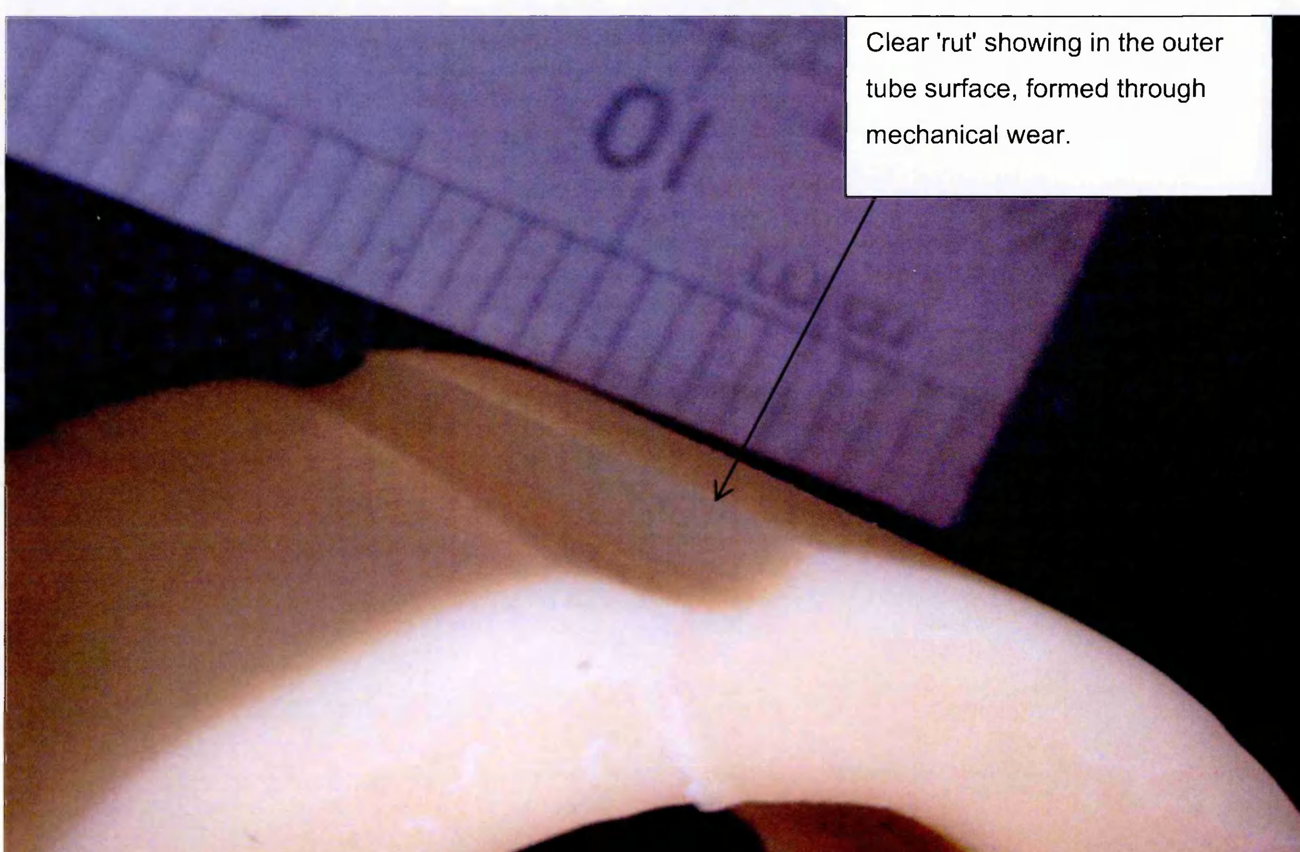


Figure 32 - Section 2 through an EPDM/PP blend tube showing damage due to mechanical wear

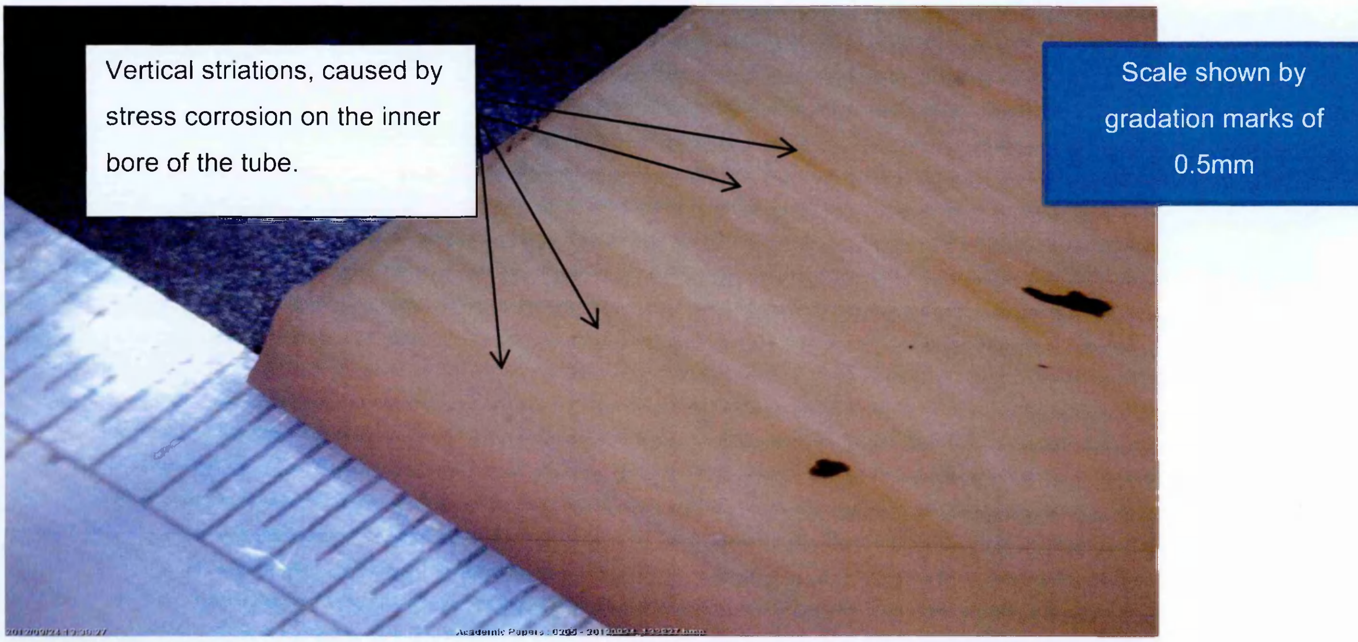


Figure 33 - Stress Corrosion Damage for an EPDM/PP blend tube

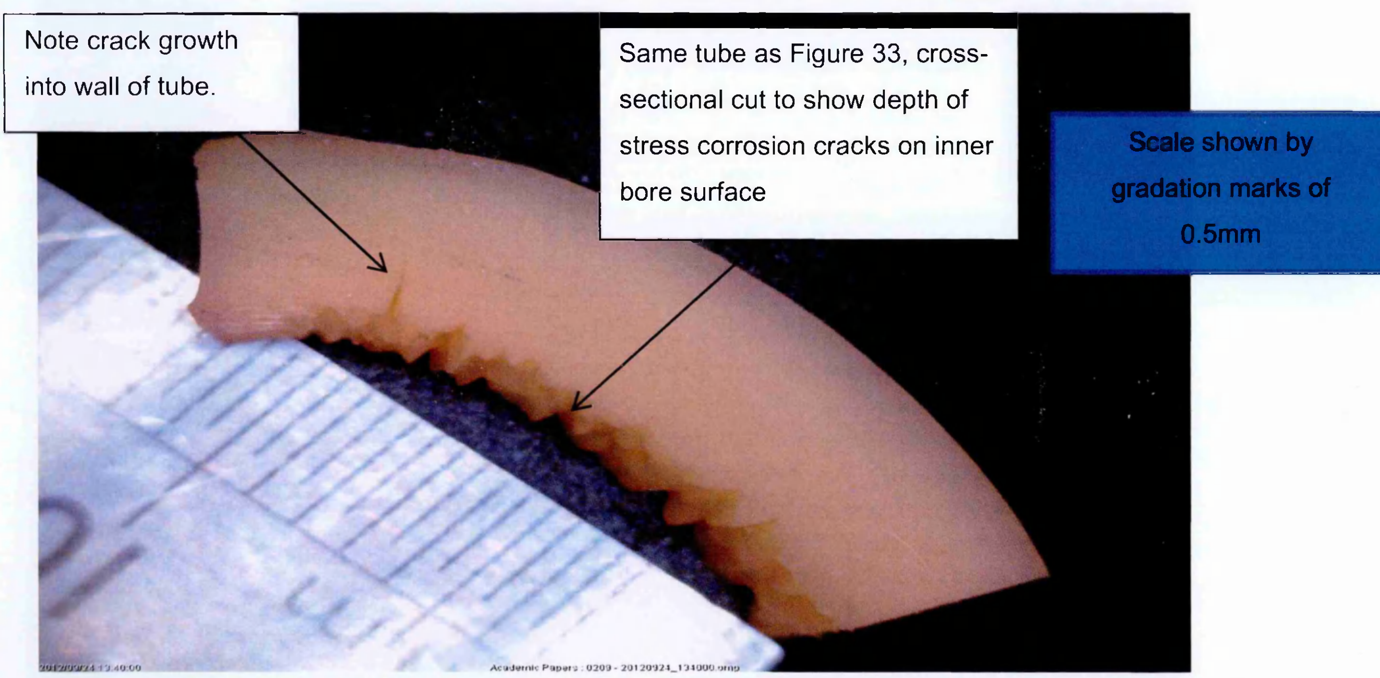


Figure 34 - Stress Corrosion Damage for an EPDM/PP blend tube

The degradation seen in Figure 33 and Figure 34 is as a result of the tube pumping for only approximately 40 hours at mean system pressures 2 bar, but with peaks at 4 bar, at a temperature of 40˚C, while pumping sulphuric acid, which is subjecting the material to chemical attack. Most 'abnormal' failures (defined as early failures or failures with non-standard fracture types or positions) are as a result of combinational degradation mechanisms. The study of environmental effects on crack growth has been considered by others (56) where it has been found that combinations of minor effects such as residual stresses or temperature fluctuations cause failures. Chemical degradation, where there is covalent bond breakage within the polymer chain, leading to mechanisms such as chain scission and reduction in molecular weight, can play a key role in these combinational effects. Applied stresses can dramatically accelerate the chemical degradation process, this is often termed 'environmental stress corrosion cracking' (ESC) as seen in Figure 33 and Figure 34. The chemical compatibility of the two materials considered within the study is shown in appendix 20.

This reaction combined with incorrect occlusion levels and high temperatures can result in increased levels of mechanical wear, particularly on the outer surface of the tube, see Figure 35.

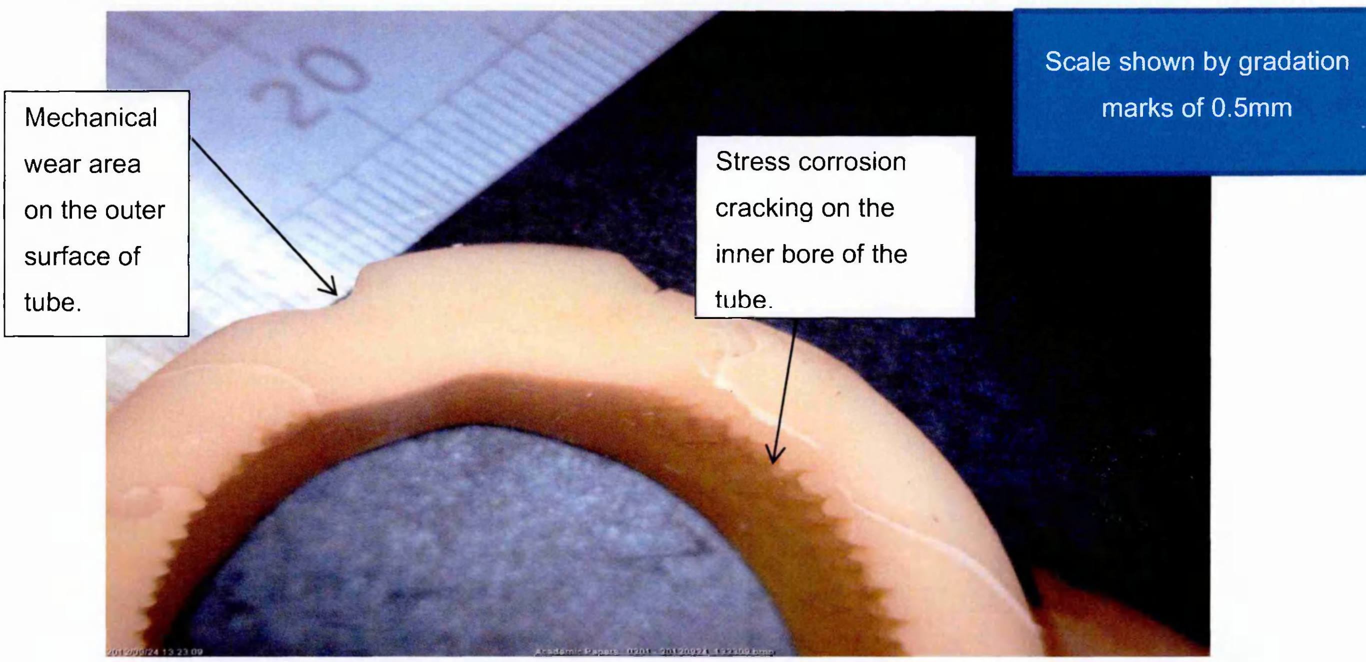


Figure 35 – Combinational degradation: mechanical wear on the outer surface of an EPDM/PP blend tube which also shows signs of ESC on the inner bore.

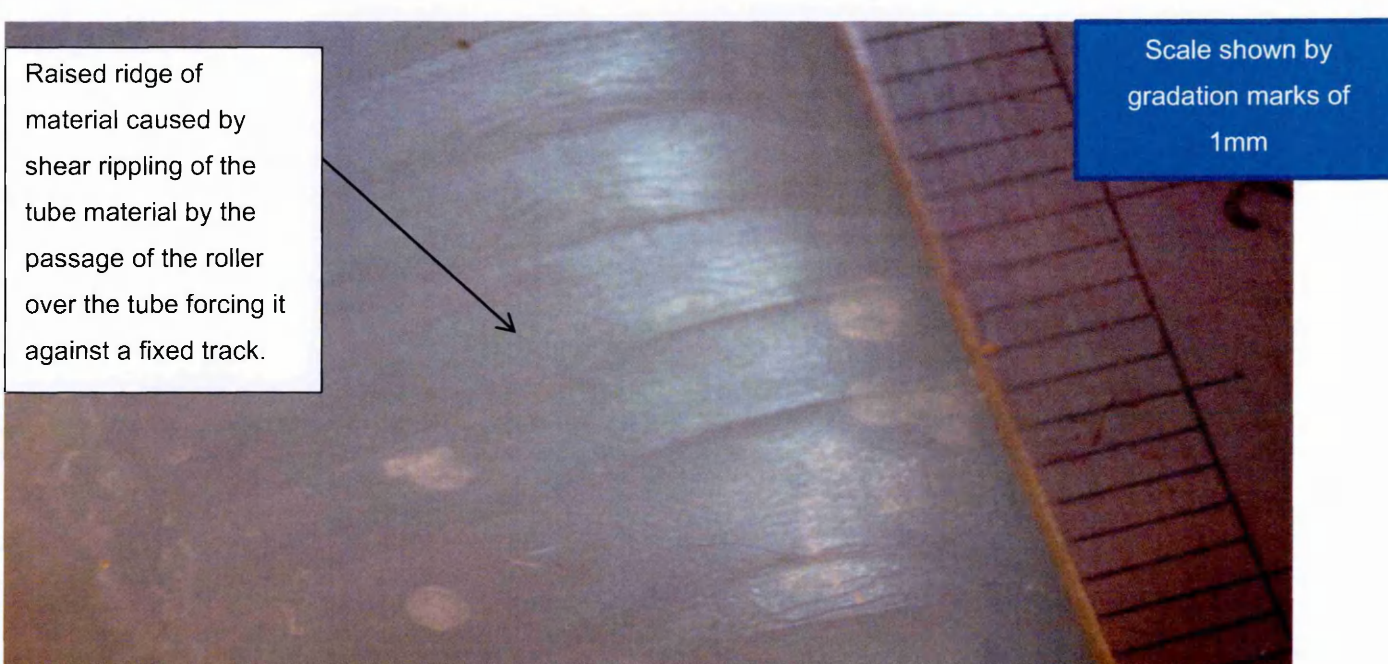


Figure 36 - Shear damage caused by roller interaction with the surface on a silicone rubber tube

Shear or frictional damage causes quite specific forms of material damage on silicone rubber tubing. This mechanism is not fully understood, it is suggested the increased frictional forces between the tube and the fixed track plus changes in the shear profile between the roller and the tube material result in localised heat build-up and material movement which gives the material the rippled appearance shown.

The complexities of the interacting failure mechanisms for all tube failures would be beyond the scope of this body of work, however from analysing interactions between factors and their effect on tube performance then it is possible to see where early failure is more likely to occur and cater for that within a prediction mechanism.

3.3 Silicone rubber

A number of hot vulcanised silicone rubbers were examined in detail to look at the complexity of linking their material characteristics to their performance as peristaltic pump tube materials, when manufactured via an extrusion process.

3.3.1 Initial data held – ' How to get a feel for the data'

The recognition of the weak linkage between the data provided by the raw material supplier and the ability to predict the behaviour of the resultant tube has been demonstrated by the work of others. A recent study (10) failed to draw good direct linkage between raw material characteristics and the performance of resultant tubing. When looking at raw material data held, it can be shown that for a silicone rubber it can be difficult to select which will perform the most consistently simply from the raw material data supplied by the material manufacturer. The test methods were comparable in nature. A number of raw material batches were reviewed for two HTV silicone type and the raw material data results were averaged; see Table 3.

Material Characteristic	**Test Method**	**Silicone 1 Result**	**Test Method**	**Silicone 2 Results**
Hardness	*DIN53505*	60	*BS903 Part A57*	58
Specific Gravity	*DIN53479*	1.15	*BS903 Part AA*	1.14
Tear Strength	*ASTMD624*	42 N/mm²	*ASTMD624*	33.8 N/mm²
Ultimate Tensile Strength	*DIN53504-S1*	11 N/mm²	*BS903 Part A2*	11.2 N/mm²
Elongation at break	*DIN53504-S1*	800%	*BS903 Part A2*	574%

Table 3 - Table of average raw material data for two comparable silicones

The table shows there are some areas where differences occur and are indeed quite prominent, most notably the elongation at break and tear strength, but it would be difficult to select the better tubing material using just this information. These two raw materials were extruded on the same extrusion line to produce tubes of differing sizes. A population of 81 of these were then subjected to comparable performance testing on standard test stations.

Performance in a variety of pumps	Silicone 1	Silicone 2
Test SC1 – Life (hours)	747	485
Test SC1 – Q_d (% per 24hr)	0.09%	0.21%
Test SC2 – Life (hours)	186	118
Test SC2 – Q_d (% per 24hr)	0.35%	0.42%
Test SC3 – Life (hours)	405	270
Test SC3 – Q_d (% per 24hr)	0.2%	0.04%

Table 4 - Tube performance characteristics for two comparable silicones

It can be seen from the results that silicone 1 shows consistently longer life than silicone 2, see Table 4. The material also shows consistently lower levels of deformation over the life of the tube, demonstrated by the lower rate in flow drop. As explained in 2.5.4 a drop in flow is an indicator of how much permanent deformation the tube has undergone. Using the figures in Table 4 some suggested linkages can be drawn between raw material data and subsequent tube performance at this stage. The material that shows a larger elongation at break and higher tear strength indicated a material which produces tubes with a longer life. However using this single parameter as the basis for an algorithm design is not a resilient approach. A predictive algorithm needs to identify other key aspects of the material that bring consistent performance when in a pump.

3.3.2 Using the key material indicator tests

Techniques for the analysis of silicone rubber are put forward in the work of others (57) (58) (23) (59) (22). It is suggested that a number of 'key indicator tests' can be used to allow differentiation between similar silicone rubbers to be used as tube materials, allowing links to be drawn between network structure and subsequent performance as tube materials. These tests were commissioned by the author at TARRC (Tun Abdul Razak Research Centre)

3.3.2.1 *Key Indicator 1 - Total extractables*

Total extractable levels for two silicone rubbers were performed using the methodology outlined by the US food and drug administration test 21CFR177.2600. This is a common test used on silicone rubber by users in industry, whereby test articles undergo extraction in n-hexane for seven hours followed by drying, the residue being determined gravimetrically.

	Silicone 1 result	**Silicone 2 result**
Total Extractable	1.812 mg/cm²	2.25 mg/cm²

Table 5 - Total extractable levels for two silicones

It can be seen that silicone 1 shows 25% less total extractable material compared to silicone 2, see Table 5. This suggests that the level of free chains available for extraction is greater in silicone 2 than in silicone 1. Using the lower level of extractable material in silicone 1 as an indicator, it can be suggested that the network structure formed in silicone 1 has formed a greater number of bonds throughout its bulk, leaving less unbound material free for extraction. The formation of a greater number of bonds, both between the polymeric chains and between the polymer and the silica filler, would suggest a more effective network structure capable of resisting fatigue to greater extent than a network structure with more unbound material.

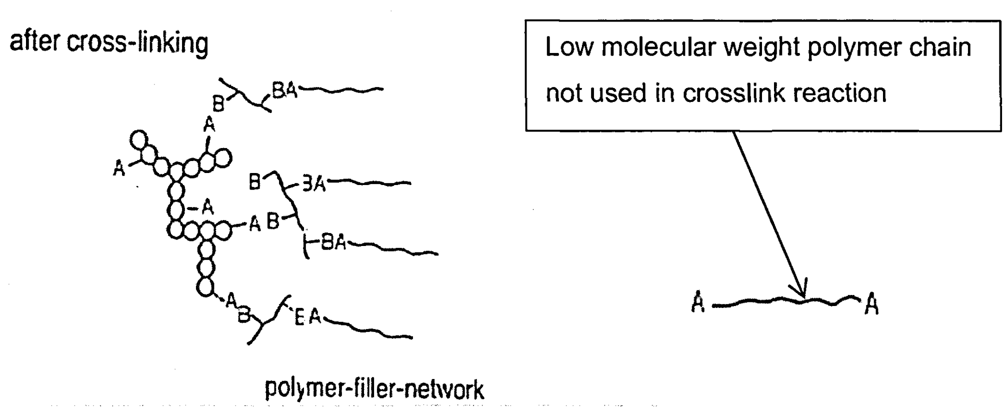


Figure 37 - Unbound chains not used in reaction (13)

3.3.2.2 *Key Indicator 2 - Swell testing*

The network structure for each silicone can be explored further with the use of swell testing, detailed in chapter 2. The two silicone rubbers subjected to the total extractable test underwent swell testing, see Table 6.

	Silicone 1 result	Silicone 2 result
W_o	0.37g	0.35g
W_s	0.78g	0.81g
SR	1.1081	1.3142

Table 6 - Swell testing results for two silicones

The greater swell ratio in silicone 2 is consistent with a lower level of crosslinking within the material. Silicone 1 shows a lower level of swelling, showing correlation with a more effective network structure and collaboration with the lower levels of total extractable data seen in the hexane extraction test.

3.3.2.3 Key Indicator 3 – Using key volatiles obtained by Headspace-GC

When subjected to more a detailed extraction both on and in the tube, using head-space gas chromatography, the two silicones show differing results. Tubing samples are produced in both materials but are not subjected to a post-extrusion heating cycle. Since the post extrusion heating cycle, called post-bake, is used to drive off volatile cyclic silicone fragments, by removing this operation it is possible to see the changes that occur when the material is subjected to different treatments. The samples are heated to 180˚C in a capped glass vial for 40 minutes. All volatile compounds, silanes and siloxanes adsorbed onto the surface of the tubing or absorbed within the tubing material, are released into the headspace. This is then transferred and injected into a Gas Chromatography Mass Spectrometer (GC/MS) for identification. The gas chromatography uses a capillary column which depends on the columns dimensions and phase property. The difference in chemical properties between different molecules in a mixture, allows the separation of the molecules as the sample travels the length of the column. They therefore leave the column at different times. Mass spectrometer is then used to identify the molecules. Combining the two processes reduces the identification errors as it is unlikely that two different molecules will behave in the same way in both GC and MS.

One of the volatiles isolated within these extraction experiments shows promise as an indicator of a silicone material which performs well in a peristaltic pump. Octamethylycyclotetrasiloxane (OMCS) showed discernible levels of difference between the two materials. This pattern held regardless of the extrusion process used to produce the tube, always being lower in the better performing tube, see Table 7.

Extrusion process ID	Silicone 1	Silicone 2
Process 1	1100 µg/g	1200 µg/g
Process 2	1200 µg/g	1600 µg/g

Table 7 - Levels of OMCS in two silicone rubbers

This volatile also shows key changes when the materials were subjected to irradiation at a 50kGy dosage, this dosage being typical of the sterilisation dosage used across the pump industry.

Extrusion process ID	Silicone 1	Silicone 2
Process 1	0 µg/g	300 µg/g
Process 2	-580 µg/g	100 µg/g

Table 8 - Changes in OMCS levels after 50kGy of irradiation

In both extrusion cases silicone 2 showed an increase, whereas silicone 1 shows no change or a decrease, see Table 8. It has been known for some time through the work of others that irradiation can be used for crosslinking (60) and a decrease in the level of this volatile could indicate an increase in crosslinking in the material: this is shown to be true for silicone 1 and the performance figures over a population of 36 match this, see Table 9.

Test ID	Irradiation level	Average Life - hours	Average flow drop %/24hrs
IR1W600	No Irradiation	124	1.44
IR2W600	50kGy	301	0.2

Table 9 - Change in performance of silicone 1 as a result of irradiation

Studies by others (61) have shown that cyclic polydimethylsiloxane species within the range D_4 to D_{18} (where the subscript refers to the number of Si-O bonds within the structure) dominate the extractable matter in room temperature vulcanised (RTV) siloxanes and that thermal ageing at elevated temperatures may cause a build-up of OMCS in a polymer matrix. However, this is shown not to be true for all silicones, whether they are RTV or hot temperature vulcanised (HTV) and indeed for the HTV silicone that performed better within a peristaltic pump, it is a species which is reduced when subjected to thermal ageing; as seen in silicone 1 in Table 10.

Extrusion process ID	Silicone 1	Silicone 2
Process 1	-340 µg/g	100 µg/g
Process 2	-340 µg/g	200 µg/g

Table 10 - Change in OMCS levels after thermal ageing @ 123˚C for 2 hours

Indeed, when subjected to thermal ageing silicone 1 shows a reduction in all cyclic species found. This could be influenced by the experimental method which calls for the analysis to be carried out before the post bake operation. This section of this key indicator method should therefore be used as a comparable test only.

3.3.2.4 Other factors to consider

Taking these two silicones and looking at their silica loading and silica type, supplied by the raw material supplier in approximated form, silicone 1 has 30% loading and silicone 2 has a 22% loading. The key indicator test and the performance data gathered suggests that the higher silica loading leads to a more effective network structure. This has been highlighted by numerous studies (59) (62). It is believed this effective network structure has also been enhanced by the development of a specific silica structure in silicone 1, which offers, through its porous nature, not only network entanglement but additional bonding sites. This is highlighted by the lower levels of low molecular weight extractables, lower levels of swelling and its improved tube performance over silicone 2.

The work by others (63) has studied how gamma irradiation can be utilised to improve the crosslink density. This is generally considered to improve the mechanical properties of silicone rubber. However, although it has been shown that the life performance within the peristaltic pump is improved by a 50kGy irradiation dosage, see Table 9, it is also shown that the level of improvement is highly dependent on both the raw material used and the extrusion process it is subjected to.

Work by others (64) has shown that the level and type of silica fillers within a silicone rubber will have a significant influence on the rate of crosslinking that can occur under irradiation conditions. Different raw materials with differing silica levels and silica structures will therefore differ in their influence on the effect irradiation on a material. It is also known that irradiation induces further crosslinking between the silica particles and the PDMS bulk. Therefore the dispersion and size distribution, brought about by the extrusion process, will also significantly affect the influence of irradiation.

3.3.3 Conclusions

By comparing raw material data with the performance data of a variety of different size tubes, produced from that material it is possible to gain a 'feel for the data'. Subsequent indicator tests can then be used to build a more detailed picture of the materials network structure. These results can give an indication of how the material is likely to perform against similar materials when exposed to gamma irradiation or thermal ageing. They can also be used as a part of the assessment of one extrusion process versus another.

Results show that the silicone material with the higher silica loading and more effective silica structure forms a network structure with high levels of crosslinking, which is more resilient to fatigue and shows lower levels of deformation over time. The importance of the network structure effectiveness is apparent, in particular, in the links between the low levels of un-bonded low molecular weight, entangled molecules and lower deformation levels.

This is shown respectively, by the silicone materials which display both a lower levels of extractable and lower values of flow drop over time. When comparing comparable silicone materials, a network structure that shows consistently low levels of deformation change has also proven most effective in resisting fracture, which differs quite markedly with some previous material studies (10). It has also shown this is a consistent characteristic and is hence predictable in terms of use for an algorithm.

3.4 EPDM / PP Blends

A number of EPDM / PP materials were examined in detail to look at the network structure formed when they were manufactured into peristaltic pump tubing using an extrusion process.

3.4.1 Initial data held

Looking at initial data for two EPDM/PP blends of similar hardness, see Table 11, it is difficult to pin point certificate values which could be easily associated with good pump performance. There are slight differences in a number of characteristics, tear strength, ultimate tensile strength, modulus at 100% elongation and elongation at break. This data suggests that blend 2 may perform slightly better, with slightly higher values in all test categories.

	Blend 1 Results / Test Method	Blend 2 Results / Test Method

Compression set	35% ASTM D412	36% ASTM D412
Ultimate Tensile Strength	15.5 MPa ASTM D412	17.6 MPa ASTM D412
Elongation at break	560% ASTM D412	580% ASTM D412
Modulus at 100% elongation	6.80 MPa ASTM D412	7.10 MPa ASTM D412
Hardness	94 Shore A	93 Shore A
Tear B	45 kN/m ASTM D624	54 kN/m ASTM D624

Table 11 - Initial data held for two EPMD/PP blends

Material from both blends is extruded into tubing of the same size, a population of 36 tubes is then tested to analyse the baseline performance in terms of average life and flow drop per 24hrs, see Table 12.

	Blend 1	**Blend 2**
Average Life	199	306
Average Q_d (% per 24hr)	3.4	0.5

Table 12 - Average performance for tubes produced from two EPDM/PP blends

Comparing this with the performance of the tube extruded from the materials we can see clear differences in life and flow drop, although the slight difference observed in the raw material certificate value could be linked to performance at this point, these links are not substantial enough to offer the robustness needed for prediction of performance.

As with the silicone material it is necessary to look at key material indicators to understand the material characteristics needed to achieve good peristaltic pump performance.

3.4.2 Using the key indicator tests

Techniques for the analysis of EPMD/PP blends are put forward in the work of others (65) (28) (26) (66).It is suggested that a number of 'key indicator tests' can be used to allow differentiation between EPDM/PP blends to be used as tube materials, allowing links to be drawn between the material structure, how the extrusion process may affect it and the subsequent performance as a tube material. These tests were commissioned by the author at TARRC (Tun Abdul Razak Research Centre)

3.4.2.1 Key Indicator 1

Portions of the tube are cut and pressed against germanium crystals then analysed using FTIR, Fourier Transform Infrared Spectrometry. Like all infrared spectroscopy techniques it relies on the fact that no two unique molecular structures produce the same infrared spectrum. The FTIR method is one which is able to measure all infrared frequencies simultaneously, rather than individually, making it faster than other spectroscopy techniques. The use of a mathematical technique, Fourier transformation, allows a computer to decipher the frequency spectrum and make identification. Using this technique on the EPDM/PP blend samples the level of silica and crystalline PP is recorded and compared for the two materials. The results show that material 2 has slightly higher levels of silica filler and crystalline PP, see Table 13.

Blend ID	Silica	Crystalline PP
Material 1	0.4	0.21
Material 2	0.5	0.24

Table 13 - FTIR measurements for two EPDM/PP blends

3.4.2.2 Key Indicator 2

Additional samples were taken of the two tubes and heated on TGA to 800˚C in nitrogen and the atmosphere changed to oxygen at 800˚C. TGA, thermo gravimetric analysis, is a method in which changes in physical and chemical properties of materials are measured as a function of increasing temperature (with a constant heat rate).The method can also be measured as a function of time (with constant temperature and / or constant material loss). Sequential FTIR spectra were obtained for the evolved gases from decomposition.

Blend ID	Initial Losses from non-rubber (wt %)	Main Polymer loss EPDM & PP (wt %)	Residual ash	Early stage CH_3/CH_2	Main loss ethylene/propylene
Material 1	24.7	64.9	9.7	0.5	0.66
Material 2	27.8	62.7	9.5	0.44	0.76

Table 14 - TGIR weight losses and FTIR ratios for two EPDM/PP blends

The key differences observed using both the FTIR and TGIR methods are:

- Material 2 has slightly higher filler levels, the filler being silica based
- Material 2 has slightly higher levels of Polypropylene
- Material 2 shows slightly higher initial losses from non-rubber contents, which suggest higher levels of oils

The results are quite close in value, see Table 14, so repeat runs of the experiment could be carried out to improve the confidence level of the figures. However these early indicator tests begin to allow links to be drawn between certain material properties and the performance of the tube produced from them. Further indicator tests help to clarify the reasons and mechanism behind why one material performs better than another within a peristaltic pump.

3.4.2.3 Key Indicator 3

Differential scanning Calorimetry, DSC, can be used to look at the amount of polymer mobility within samples tested. DSC is a thermo analytical technique in which the amount of heat required to increase the temperature of a sample is measured. It works on the premise that as a sample undergoes a physical transformation, such as a phase transition like crystallisation, more or less heat will need to flow to maintain the sample at the same temperature. Checking the heat flow of a sample against a reference allows DSC to measure the amount of heat absorbed or released during such transitions.

Portions of tube are cut and heated for 200˚C @ a rate of 20 ˚C / minute and then cooled at the same rate.

Blend ID	**Area of crystallisation**
Material 1	48.8 J/g
Material 2	50.0 J/g

Table 15 - DSC Area of Crystallisation results for two EPDM/PP blends

The thermal energy needed to mobilise a constrained polymeric chain is higher than an unconstrained polymeric chain. Material 2 therefore shows a slightly higher level of constrained polymer than material 1, see Table 15; this suggests perhaps a higher level of cross-linking within the EPDM phase of the material. This could contribute to its ability to resist fatigue when used as a material for peristaltic pump tubing. However the difference is slight so could be within experimental tolerances. The experiment could be repeated several times to assess whether the results correlate with each re-run of the experiment.

3.4.2.4 Key Indicator 4

These techniques can be used to demonstrate general material characteristics, fillers used and thermal behaviour. However, using xylene extraction to extract the polypropylene phase from the blend and then swelling the extracted samples in toluene allows the level of crosslinking within the EDPM phase to be ascertained. Samples were also extracted in hot acetone, then dried and swollen in d-chloroform before being examined by H NMR. H Nuclear magnetic spectroscopy is a method which utilises the magnetic properties of the ^{1}H atomic nuclei. This type of spectroscopy uses nuclear magnetic resonance, where a specific resonant frequency can be linked to a particular substance. On that premise it can be used to determine the physical and chemical properties of atoms or the molecules in which they are contained.

Using this method the level of Polypropylene is estimated between the total xylene extract and the acetone extract.

Blend ID	**Estimated PP content (Xylene – Acetone)**	**V_r – volume fraction in Toluene**	**NMR peak width at half height**
Material 1	22.1	0.2559	0.88
Material 2	26.1	0.2635	1.29

Table 16 - PP content and swell results for two EPDM/PP blends

Material 2 shows higher PP content and higher levels of crosslinking within its rubber phase, see Table 16. It is suggested that we now see some consistent characteristics that can be associated with improved peristaltic pump performance. The higher the levels of silica, greater crosslinking and greater levels of constrained polymer chains are displayed by the material which performs better within the peristaltic pump. The increased crosslinking within the EPDM phase offers greater resistance to fatigue. The energy imparted by the impact of the roller on the material is absorbed by the enhanced network structure which has been brought about by the higher levels of bonding. The silica particles, although unlikely to be chemically bonded to the rubber as in silicone network structures, do offer reinforcing properties to the material. It is noted that the results from the experiments are quite close so repeat runs would help improve confidence levels in the conclusions.

It is necessary to look at the key material indicators to understand the key material characteristics needed to achieve good peristaltic pump performance. This information can then be carried forward into the manufacturing process to give more consistent peristaltic pump tube. This makes life prediction easier to model.

3.4.2.5 Key Indicator 5

Work by Montoya et al (26) has shown that analysis by transmission electron microscopy can help show the microstructure of the blends. TEM is a microscopy technique whereby a beam of electrons is transmitted through an ultra-thin specimen, interacting with the specimen as it does so. This interaction allows the formation of an image. The method differs to scanning electron microscopy where a focussed beam of electrons allows interaction with the surface of a specimen; the TEM technique transmits through a specimen not just its surface.

Ultra-thin sections of sample were prepared by cryo-ultramicrotomy and collected on gold TEM grids with the aid of ethanol. Staining with ruthenium tetroxide then allows the EPDM to be shown clearly in relation to the PP phase.

The ruthenium tetroxide stains the EPDM so it appears as dark grey areas, with the PP matrix as pale grey. The fillers within the blends appear as the darkest features.

The samples give a 'snap shot' of the phase structure but if a number are carried out across the structure then an averaging of typical features can be obtained.

TEM samples were taken of both blends, Figure 38 and Figure 39.

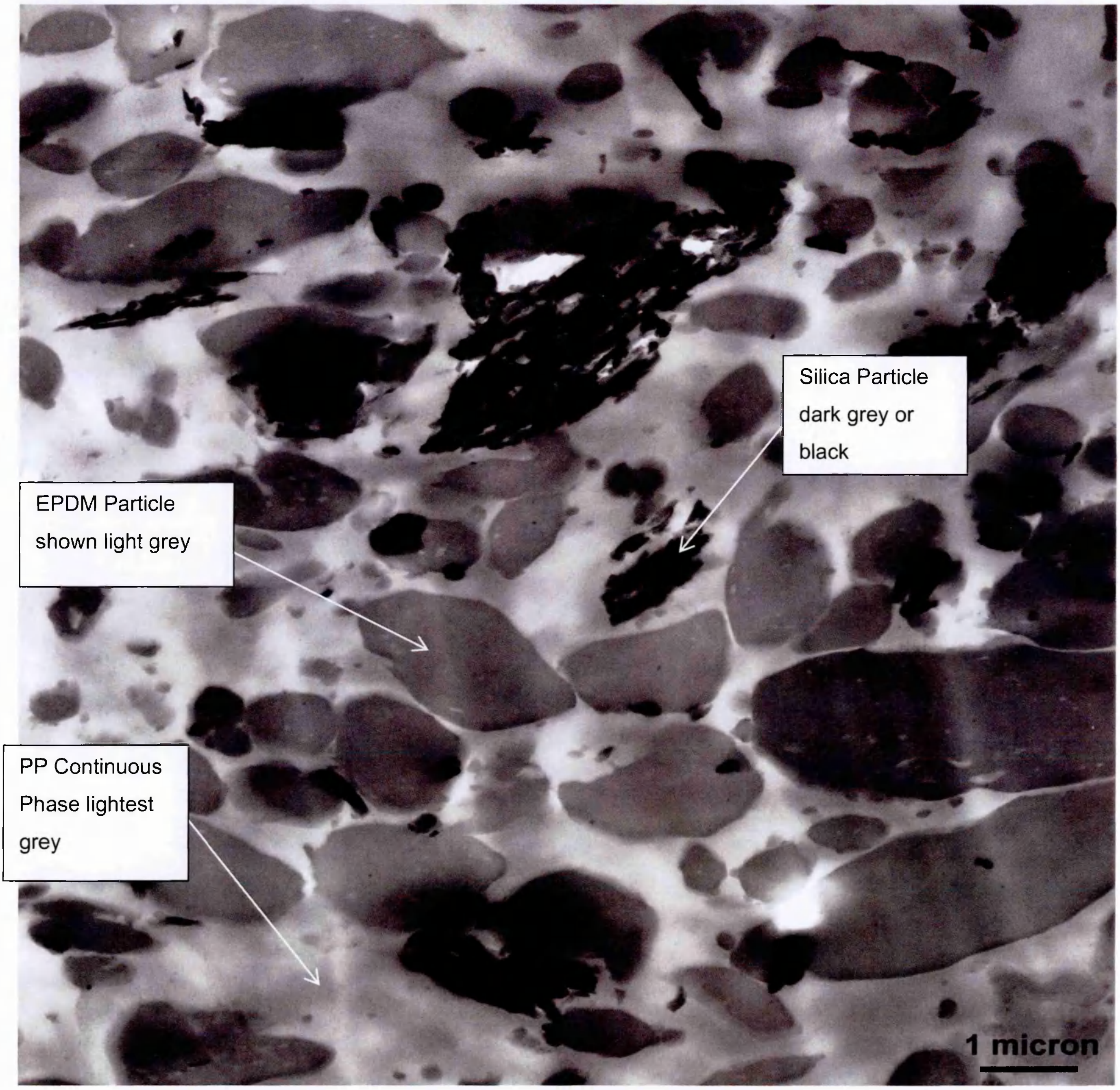


Figure 38 - TEM of Blend 1

It can be seen from both Figure 38 and Figure 39 that the structure of both tubes seems similar. The EPDM particles in blend 2 seem to be on average slightly smaller. However these images are only snap-shots of the material structure, as mentioned previously, they should be therefore be used as indicators in conjunction with the key indicator tests carried out. With a greater level of sampling throughout a sample then a better picture of structure could be built up. However, the technique can prove to be costly and the use of multiple samples needs to be assessed as to whether it provides enough clarity of the phase structure to justify the costs of multiple sampling.

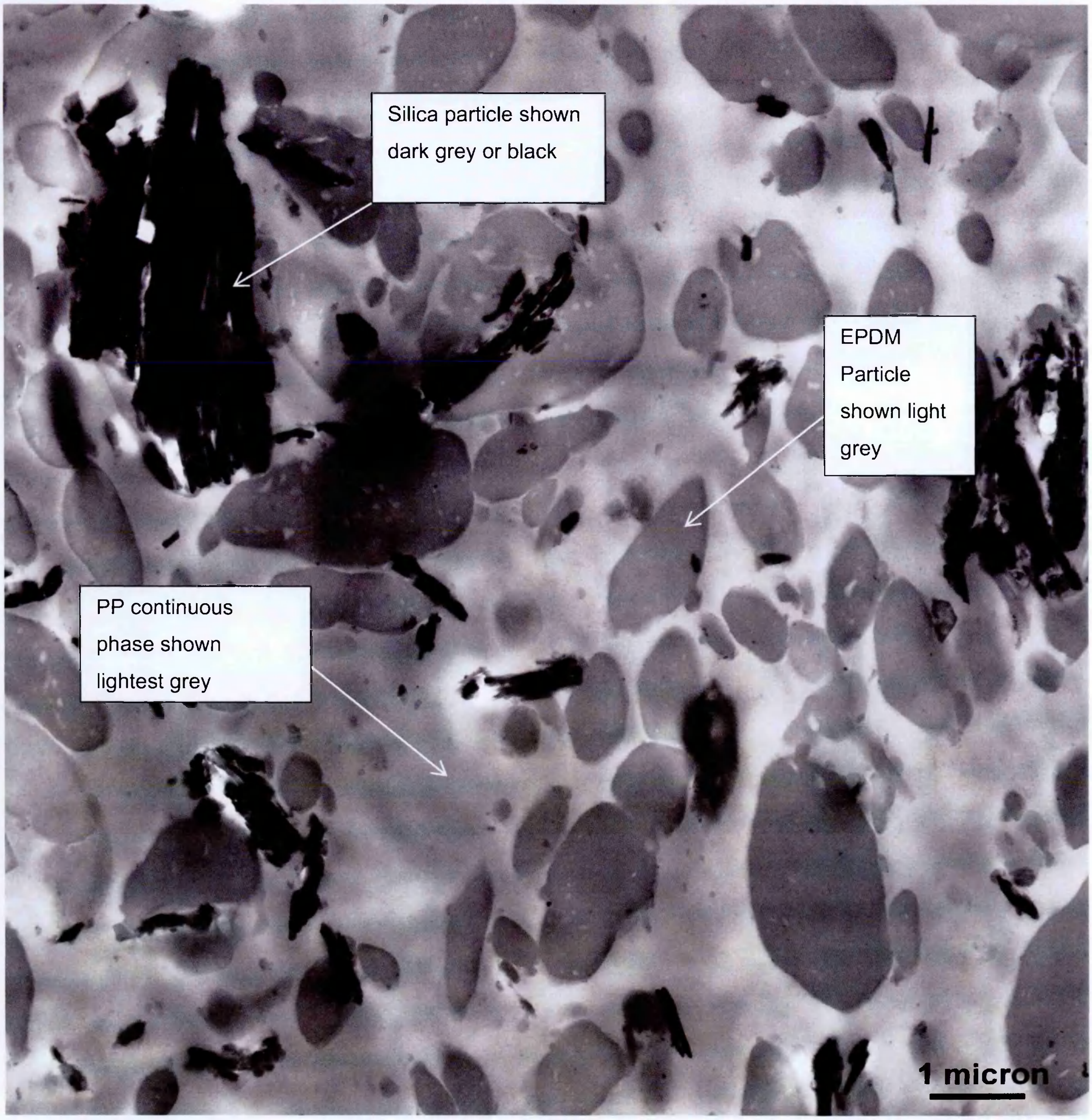


Figure 39 - TEM of Blend 2

3.4.3 Looking at the mechanism of mechanical wear for an EPDM/PP blend

Tube material which has been mechanically worn from the tube bulk during use in a peristaltic pump was analysed (67). The worked material showed clear signs of PP being lost from the material bulk, thus increasing the importance of the integrity of the EDPM phase. Therefore those materials which show a greater level of crosslinking within the EPDM phase are more resilient to fatigue, particularly if mechanical wear is present. The analysis of changes in material that are present as a result of repeated occlusions such as seen above, may offer a route for a more detailed use of the data in the development of an algorithm.

It has also been observed through empirical evidence that the oil levels within an EPDM/PP blend play an important role in the material behaviour as a peristaltic pump tube. Within certain sections of the water industry, leaching of hydrocarbon oils is a concern. The author observed an EPDM/PP blend with much lower levels of oil than shown in these samples (material 1 and material 2) demonstrated life results of just 20% of the life observed for these materials. Work by others (68) has shown that the distribution of oil through EPDM/PP blends can be complex, although the oil tends to have a preference for the EPDM phase. Its effect on the performance of a material as a peristaltic pump tube is also likely to be complex, especially when mechanical wear occurs and the EPDM phase becomes more influential in the strength of the network structure. It has been demonstrated that lower levels of oil can have a significant effect on life, therefore the indicator tests should be used to ascertain levels when evaluating a material's suitability for prediction.

3.4.4 Conclusions

The results suggest that the levels of filler, levels of oil and the cross-linking within the EPDM phase play an important role within the blend when relating the blend characteristics to tube performance. The key indicator tests show that for materials of similar PP levels the morphology effects of the EPDM crosslinking, the oil levels and the level and distribution of filler are the biggest influences on the performance of the material as a tube within a peristaltic pump. However, since the TPVs are basically melted and reformed when extruded, the extrusion process can therefore affect the end performance of the material considerably and it is this manufacturing process which is considered in detail in the next chapter.

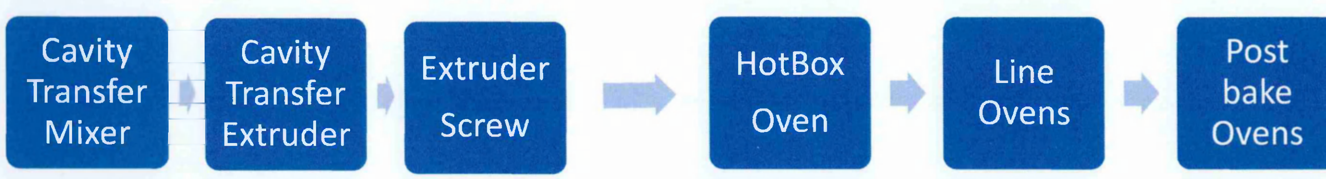
4 MANUFACTURING PROCESS

4.1 Overview

The extrusion processes used to extrude silicone rubber are quite different to those used to extrude a thermoplastic elastomer. Although both are continuous processes which make use of an extruder screw and a pin and die arrangement to form the tubing, the differences between the two are dramatic.

4.2 Silicone Rubber Extrusion

4.2.1 Overview



4.2.2 Mixing – CTM/CTE

In the extrusion of silicone rubber a cavity transfer mixer (CTM) and cavity transfer extruder (CTE) are used, both are based on a similar design although one feeds directly into the extruder screw cavity. The Cavity transfer mixer in this system is a continuous dynamic mixer and uses a twin screw mixing process to mix the material. The mixing process needs careful control. The blending of raw material and catalyst must result in a homogeneous mix. At the same time the amount of energy added to the mix needs to be controlled since local heating results in premature curing and the production of unwanted gels.

The CTM used is pictured in Figure 40; the CTE is very similar to this but feeds directly into the extruder screw. The importance of silica to the effectiveness of a silicone rubber network structure has been shown during material analysis discussed in chapter 3 and through a number of studies (59) (23) (58). In order that silica can form efficient mechanical reinforcing and bonding, it is acknowledged that it should be distributed throughout the rubber-bulk evenly. This gives a greater chance of adsorption of PDMS on the silica surface and the creation of both intra-particulate and inter-particulate connections. Also it has been noted by others

(62) that the particle size distribution considerably evolves during the dispersion, due to breakage and erosion, affecting the final characteristics of the silicone rubber.

Figure 40 - Cavity Transfer mixer

Although a tube manufacturer often has very little control over the mixing that occurred pre-delivery, it has been found that the mixing undertaken as part of the extrusion process can be optimised for the final performance characteristics of the tube produced. The mix cycles that can be applied to a raw material will alter depending on the equipment available. Some extrusion processes have more automated mixing facilities than others.
The silicone raw material is a gum type material supplied in two different groups; one group contains the catalyst and inhibitor, whilst the other does not. A silicone raw material was subjected to a number of differing mix cycles on the CTM machine shown above and then extruded under the same run conditions into the same

size tube. A population of 36 tubes were then performance tested and the life in hours and the flow drop recorded. The results demonstrate how the mix cycle can affect the performance of the tube produced.

CTM Mix Cycle time	Average life of resultant tube (hrs.)	% flow drop per 24hrs
215s	200	2.04
415s	262	1.71
615s	170	4.25

Table 17 - The effect of mix cycle time on tube performance for a silicone rubber material

For this raw material and the size of tube produced it has been shown that a mix cycle can be adjusted to give a better performing tube. However, it has been noted that mix cycle optimisation is dependent on the raw material and the mix cycle cannot be treated in isolation from the thermal cycle applied to the material.

4.2.3 Extruder Screw, Pin and Die

The extruder screw and the pin and die design have not been examined in detail within this body of work. However, a number of observations have been made on these areas of the extrusion process, which are pertinent to the production of consistent tubing to be used in a prediction mechanism.

The build-up of cavitation on entry to the pin and die area is a source of air and gas entrapment within the silicone rubber, as seen in Figure 41 where a silicone rubber sample has been removed after a line stoppage. This cavitation is as a result of fluid velocity differences between the die wall and the main body of the material.

Flow changes are also observed at the entry to the extrusion screw from the CTE, where angle changes result in distinct flow changes. Air entrapment caused by inconsistency in the feed mechanisms also contributes to instabilities in flow. The work of others (69) has highlighted that flow instabilities are an important limitation in most polymer processing operations, in extreme cases leading to melt fracture; however in this study flow characteristics lead to high levels of bubbles within the finished material.

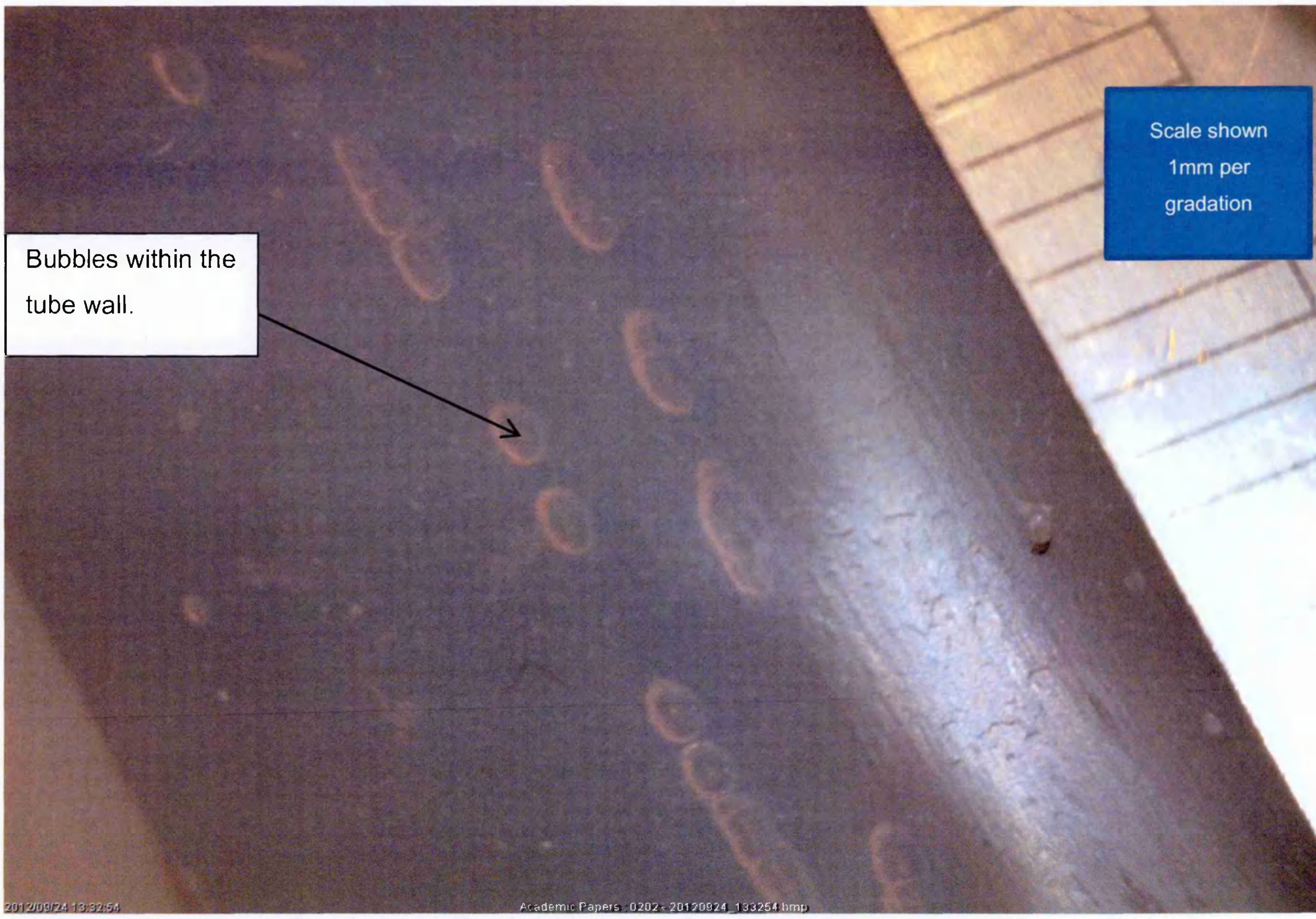


Figure 41 - Bubbles within 25.4mm bore, 4.8mm wall silicone tubing

When performance testing is carried out on tubes that display these processing defects they do not appear to limit performance radically, but the formation of bubbles within the material is likely to have some effect on the consistency in performance from tube to tube, being non-homogeneous in nature. These flow issues within certain sections of the extrusion process are also likely to influence the cross-linking reaction of the silicone rubber. The work of others (70) shows that at the beginning of the extrusion process the viscosity of the reactants is low and the chemical reaction is controlled by kinetics. With increasing time the viscosity of the reactants increases and the chemical reaction is controlled by both the reaction mechanism and the diffusion mechanism. Therefore the introduction of voids within the material during the screw and die stages are likely to influence the reactions that occur.

The need to control the wall thickness of a tube, to ensure consistent levels of occlusion and thus the associated stress levels, is a vital part of producing consistent tube needed to facilitate a predictive mechanism. A recent study by a peristaltic pump manufacturer (71) highlighted this importance. In silicone tubing the tolerance achieved on the wall thickness is governed in part by the extrudate swell experienced on the extrusion line. It has been found that the pin and die tooling design can have an instrumental effect on the dimensions that can be consistently achieved.

This is acknowledged by the work of others (72) (73) who state that the change in boundary conditions at the die exit means that analytical solutions are unusable, so that solving swell issues is best served experimentally, with approaches such as those observed in this study.

4.2.4 Thermal energy applied to the silicone material

The thermal energy applied to the silicone material is done so over three main areas: the hot box, the line oven and the post bake oven. The thermal profile for silicone rubber differs depending on the tube size being produced and the raw material from which it is produced. It is also important to recognise that the cross-linking that occurs is fundamentally a random cross-linking process as recognised by others (74). Indeed some (70) state that during the whole course of the silicone rubber vulcanisation and in the whole reaction field, at all times the chemical structure of silicone rubber changes with its spatial position; and in all spatial positions the chemical structure of silicone rubber changes with its vulcanisation time. The thermal profile applied to different silicone raw materials, on the same extrusion line, differs, as seen in Figure 42.

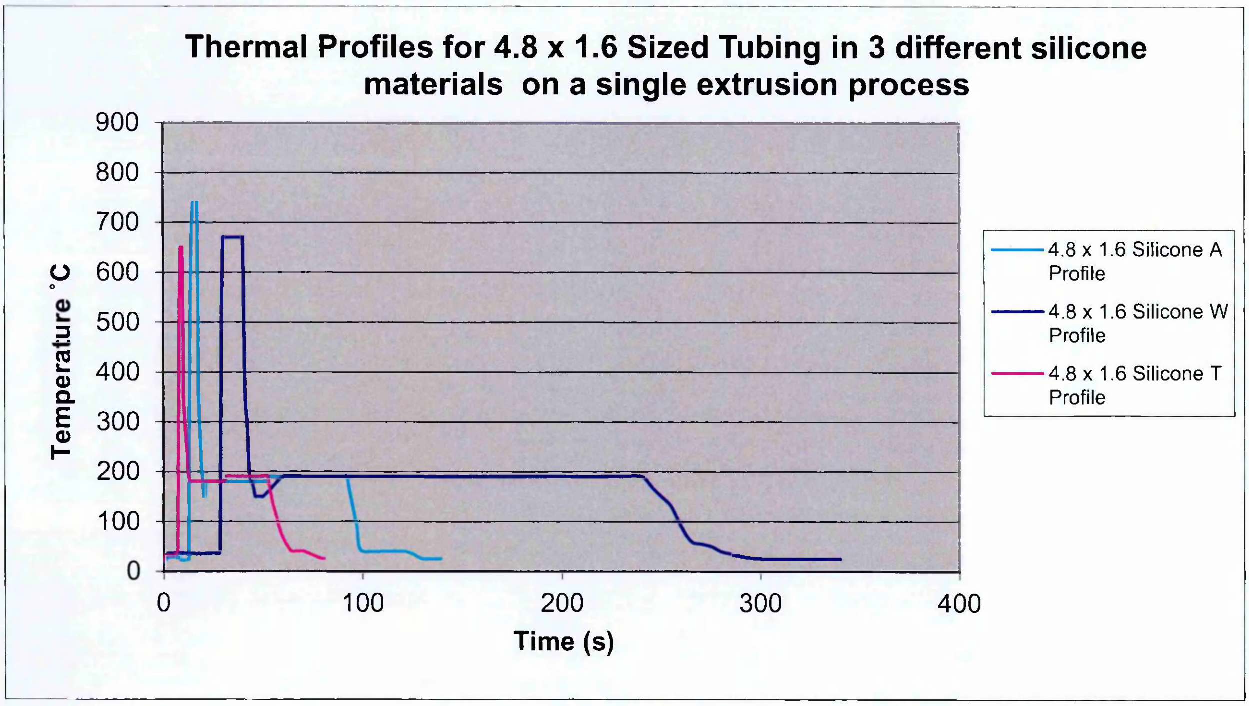


Figure 42 - Comparison of Thermal Profiles for 3 different silicone rubbers

The thermal profile for each of the silicone rubbers is quite different. It should be remembered that when these profiles are applied at the manufacturing stage, the process control could be limited to dimensional stability and tolerance and the general optical quality of the tubing being produced. Inline feedback is limited to laser measurement of the tubes' dimensions, both wall thickness and tube diameter. Additional quality checks are often limited to shore hardness and optical checks only.

When the performance results for this size tube, for these three silicones are compared over a population of 36, a clear difference can be seen, see Table 18. Silicone W shows much greater life and the greatest level of thermal energy exposure during the extrusion process. The population of tubes in this material show a much wider distribution in life, as demonstrated by the high standard deviation. Further optimisation of the thermal profile would be useful to try and reduce the spread of the population to help the use of prediction models. If the same thermal profile used for silicone W is applied to silicone A or silicone T the extrusion of the material fails, as dimensional tolerances required for the wall and bore fall outside the limits for the tube size produced. Dimensional tolerances for tube bore and wall sizes are very important to in order to maintain the correct occlusion levels within the pump head they are used in. The profile applied is therefore directly linked to the raw material being processed to ensure the greatest level of crosslinking can be achieved.

SILICONE MATERIAL ID	AVERAGE LIFE (occlusions)	STANDARD DEVIATION (occlusions)
Silicone T	2,606,400	650,296
Silicone W	6,696,000	3,075,701
Silicone A	4,248,000	945,643

Table 18 - Performance of 4.8 x 1.6 tubes in three different silicones extruded using the same process

Looking at a single material it was found that the thermal profile for different tube bore sizes but the same wall thickness varies considerably, see Figure 43.

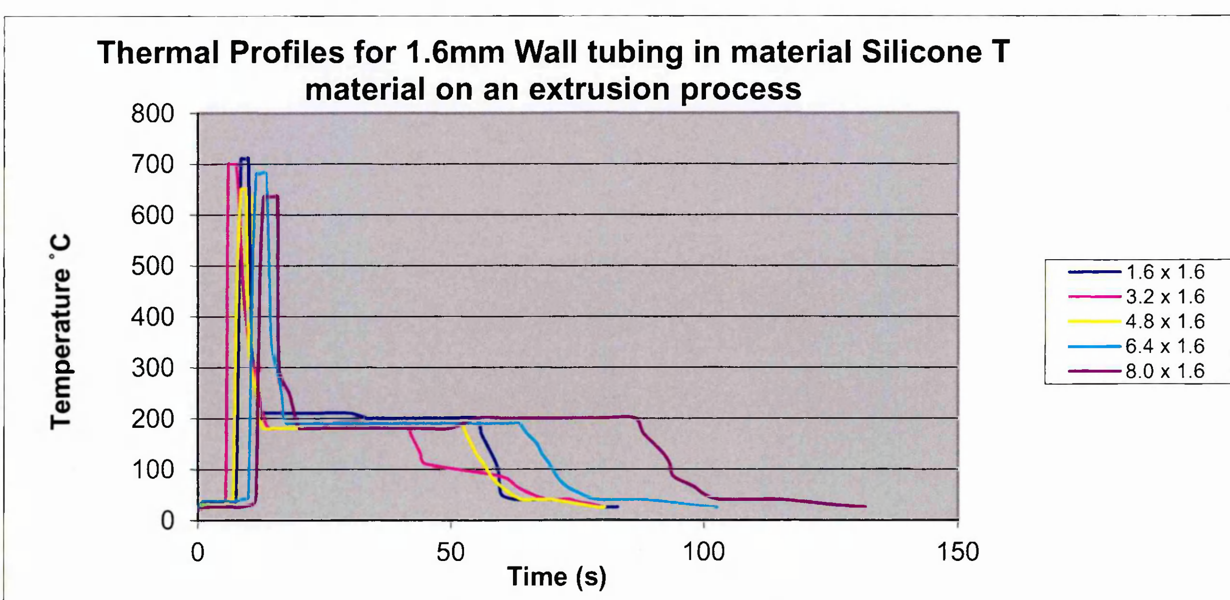


Figure 43 - Thermal profiles for 1.6mm wall tubes of different bore sizes in one single silicone material

The main reason for the difference seen is that maintaining the dimensional tolerance for tubes of different aspect ratios requires different extrusion line run rates, the result of this is different thermal profiles. Each tube size also has different surface area and volume, see Table 19. As a result each tube has a different interaction surface and material volume, affecting the chemical changes that occur during the extrusion process. Cure kinetic models have been studied in detail by others (75) and they relate to temperature and extent or degree of reaction. The amount of surface area for a tube defines the boundary at which the low molecular weight silicone component not utilised within the cross-linking reaction can diffuse out of the silicone rubber bulk. The volume of the tube defines the amount of material which requires cross-linking and the distance through which any unused components of the reaction must move before they reach the tube boundary. It will also affect the temperature rise of the reaction. Cross-linking is an exothermic reaction and HTV silicone rubbers require heat to be activated. If the temperature rises during a reaction, the reaction rate will rise to a maximum value and then return to zero as the reactant is consumed (75). The rate the temperature rises will be in part governed by the thermal resistance of the volume of material as well as the temperature applied and how efficiently the temperature is maintained.

Tube Size	Tube Bore	Tube Wall	R	r	Surface area	Volume
1.6 × 1.6	1.6	1.6	2.4	0.8	52.28 mm²	16.08 mm³
3.2 ×1.6	3.2	1.6	3.2	1.6	78.41 mm²	24.13 mm³
4.8 ×1.6	4.8	1.6	4	2.4	104.55 mm²	32.17 mm ³
6.4 ×1.6	6.4	1.6	4.8	3.2	130.69 mm²	40.21 mm³
8.0 ×1.6	8.0	1.6	5.6	4	156.83 mm²	48.25 mm³

Table 19 - Surface Area and Volume for five different tube sizes

The thermal profile applied to a material is also dependent on the extrusion process used.

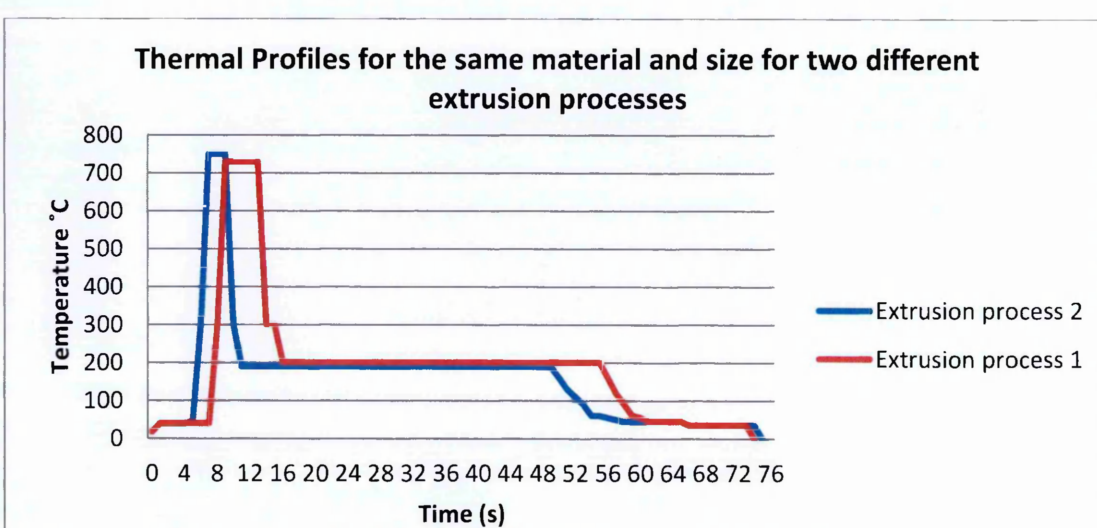


Figure 44 - How the thermal profile alters depending on the extrusion process for a 1.6 × 1.6 mm tube

The profiles shown in Figure 44 are quite different. This is due to a number of reasons:

- The extrusion lines have different designs in terms of hot box and line oven lengths, the line ovens in process 2 are longer than process 1
- In order to achieve dimensional stability the extrusion line run rates are set to suit the extrusion design
- Some parts of the extrusion line design may have different thermal capabilities or differences, i.e. process 2 has line ovens with fan assistance, however process 1 has line ovens without fan assistance.

When the performance of the tubes run under these conditions is analysed, it is clear that this results in performance differences as well, see Table 20. The results from 36 pieces of tubing are analysed, tubing from process 2 shows a 20% increase in average life and a drop in standard deviation in the population studied.

Process ID	Average Life (occlusions)	Standard Deviation (Occlusions)
Process 1	1,506,000	258,840
Process 2	1,800,000	192,000

Table 20 - Comparison of performance for 1.6 x 1.6 silicone tubes from two different extrusion processes

Process 2 produces tubing with a much more effective network structure, capable of resisting a greater number of occlusion cycles before final failure. The length of the line oven and the use of fan assistance within the line ovens is the most significant difference between process 2 and process 1. The process 2 configuration is more efficient in maintaining the correct thermal conditions for effective cross-linking.

One important part of the thermal cycle is the post bake ovens, where the tube is subjected to 2 hours at 200°C. Applying the post bake section of the thermal cycle has a significant impact on the performance of the resultant tube. Over a population of 36 tubes, the post bake thermal cycle increases the average life by 50% and reduces the standard deviation of the population by 30%.

3.2 x 1.6 Silicone Tube Process 2	Average Life (occlusions)	Standard Deviation (occlusions)
Post Bake applied to tube	2,851,200	514,272
No Post-Bake applied to tube	1,702,800	718,080

Table 21 - Comparison of life with and without post bake thermal cycle.

A silicone raw material was used to produce a single tube size over a variety of thermal profiles, see Figure 45.

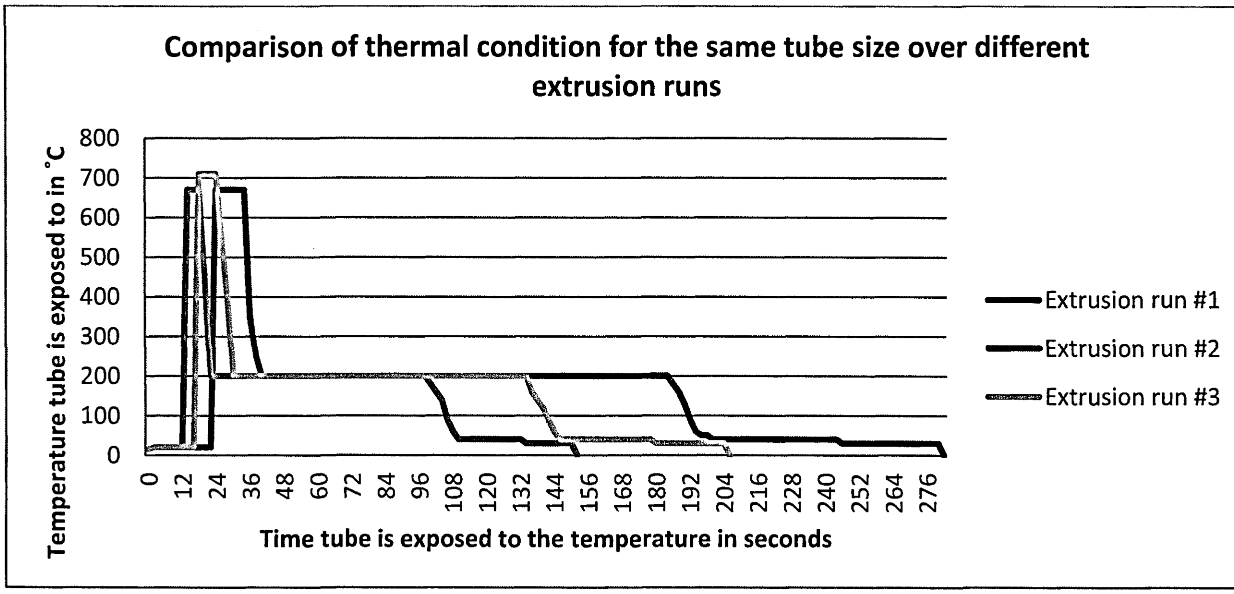


Figure 45 - Comparison of thermal condition for 3.2 x 1.8mm tubing in the same silicone material

This tube was then subjected to performance testing. To isolate how the variation in thermal cycle affected performance the performance aspect analysed was the tube's ability to resist deformation over repetitive doses. This was measured by comparing each tube's ability to repeatedly pump a set dose size, see Figure 46.

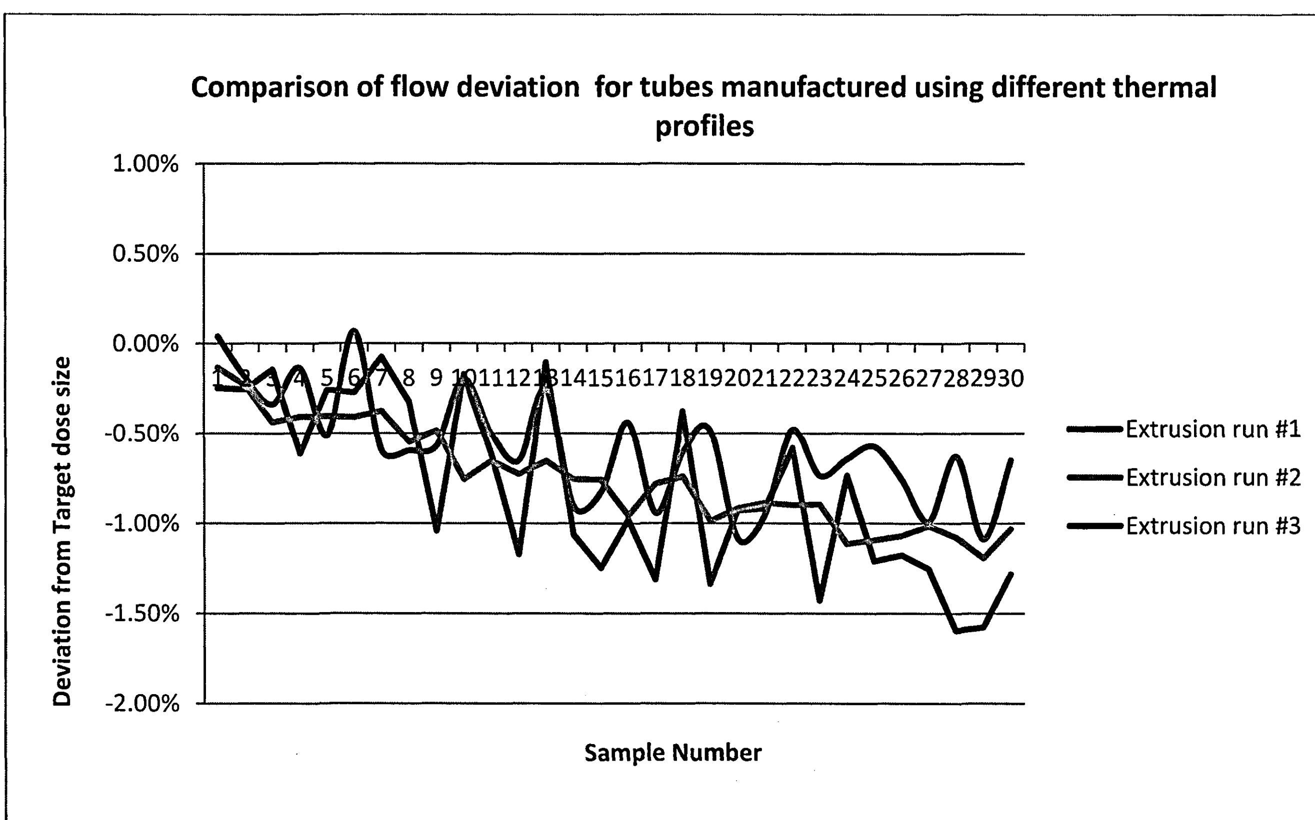


Figure 46 - Comparison of Qd performance for 3.2 x 1.8mm tubing in the same silicone material run in different extrusion conditions

It can be seen that even with quite different thermal profiles there are quite small changes in performance. The performance of tubing produced using the thermal conditions set in extrusion run #1 shows the greatest level of flow drop over the doses taken. Whilst the performance of tubing produced using the thermal conditions set in extrusion run #3 shows the lowest level of flow drop. However from a prediction point of view it is perhaps the tubing which is produced using the thermal conditions set in extrusion run #2 which are of most interest. This tubing produces a stable flow drop over the doses taken, with the least variation dose to dose. It is this pattern of behaviour which is most easily modelled within an algorithm and therefore these extrusion conditions which are most attractive from a tube life modelling point of view.

It is clear from the experiments undertaken of the difficulties faced in optimising the thermal cycle for a particular raw material and indeed for a particular size of tube. In order for optimisation to be introduced the tube material, tube size and extrusion process must all be known. Dimensional tolerances and visual checks can be used on the line but then post extrusion performance testing needs to be carried out to ensure that the profile has been optimised

4.2.5 Consideration of other processes: Injection moulding

Injection moulding is another process that can also be used to manufacture tubing used in peristaltic pumps. A small number of experiments were undertaken using prototype tooling for one size of silicone tubing, 9.6mm bore × 2.4mm wall. The injection moulding process allowed the temperature at which the tube was moulded and the time it was held at the moulding temperature to be adjusted.

A HTV liquid silicone rubber by Wacker, Elastosil LR3003 was used to produce the tubing. The tubing was produced through the use of a DOE matrix, see Table 22, to control various parameters of the injection moulding process. This DOE matrix allows 3 factors to be analysed;

- Four different cure times; the length of time the melt was held in the mould.
- Two different injection temperatures; the temperature of the melt when injected into the mould.
- Whether the tube was subjected to a post bake thermal cycle or not.

The tubing produced, a population of 64 was then subjected to performance testing and the results were analysed, see Figure 47.

	A Cure Time		B Inj Temp		C Post-Bake	
Level	4	420s - 7m		-	-	-
	3	360s - 6m		-	-	-
	2	300s - 5m		120 Deg C		Post-Bake
Run	1	240s - 4m		165 Deg C		Non Post-Bake
1	1	240s	1	120	1	NPB
2	2	300s	2	120	2	PB
6	2	300s	1	120	1	NPB
3	4	420s	1	120	2	PB
4	3	360s	2	120	1	NPB
5	3	360s	1	120	2	PB
7	1	240s	2	120	2	PB
8	4	420s	2	120	1	NPB

Table 22 - DOE Matrix to explore Injection moulding parameters for LSR

It was found that as with extruded silicone rubber, post baking played a vital role in improving the performance of the tube. The average life of a tube subjected to a post bake cycle was 5 times that of a tube which didn't have a post bake cycle. The post-bake cycle was 2hrs at 200°C (same as with the extruded tubing). The cure time did have an effect on performance, with the longer cure time giving a better performing tube in terms of life. However, during the experimentation, the injection temperature could not be explored due to process constraints, hence further experimentation would have to be done to explore this area further and check interactions between cure time and injection temperature.

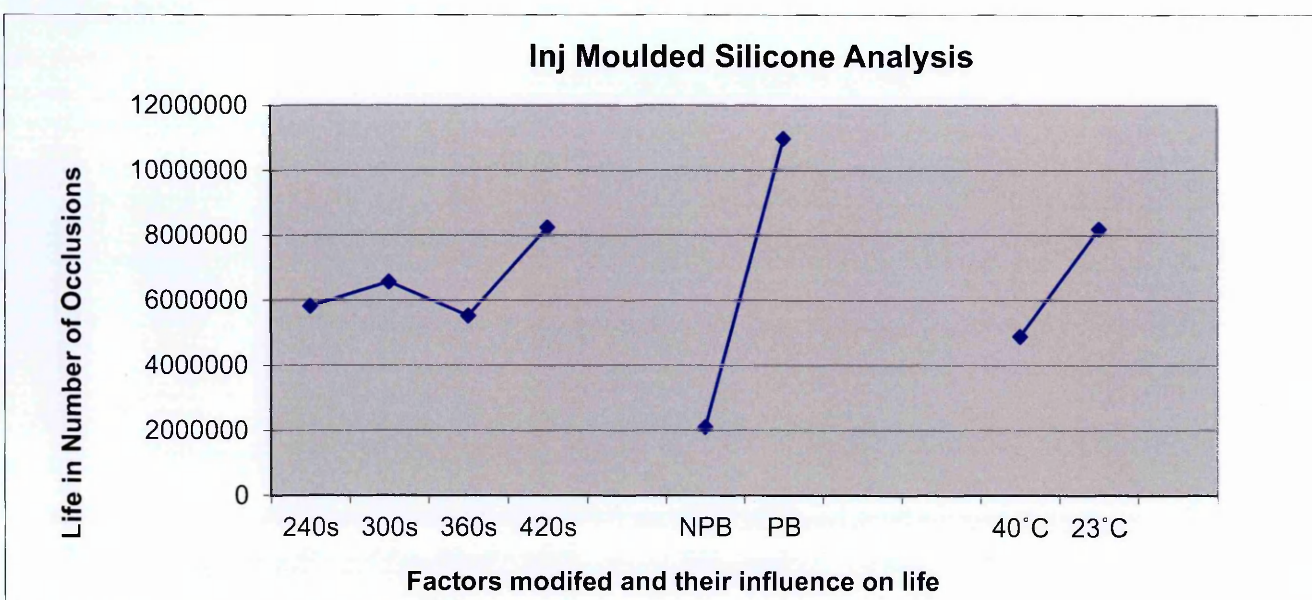


Figure 47 - The effect of process parameters (shown in table 22) on the performance of injection moulded tubing

Comparing the life of this injection moulded tube to the equivalent sized extruded tube we find that the injection moulded tube has a life in excess of 3 times of that seen in an extruded tube. It is therefore reasonable to suggest that it is worth pursuing this manufacturing technique more fully for representation in an algorithm.

4.2.6 Conclusions

It can be shown that by using different aspects of the extrusion process, a silicone rubber can be optimised for use within a peristaltic pump to give a more consistent performance from tube to tube, thus making a predictive mechanism easier to facilitate. However, the mechanism can only be developed fully when the effects of the environment on it are understood, and it is these that are discussed in chapter 5.

4.3 TPE Extrusion

4.3.1 Overview

4.3.2 Extruder screw speed and the effects on material properties

The variables that can be altered on a TPE extruder line, used for EPDM/PP blends, vary considerably to those on the silicone tube extrusion line. It has been found that on the extrusion line studied there is one key variable which has most influence on the properties of the tube produced from it and this is extruder screw speed.

To look at the effect of extruder speed in detail; one grade of raw material from one material supply was exposed to different extruder speeds, to produce both tube and flat sheet. Initially extruder speed limits were found by noting the results at very low and very high extruder speeds. Where the material was not mixed or melted properly, it suggests an extruder speed that is too low. Where melt fracture occurs, it suggests an extruder speed which is too high. The extruder speeds chosen were therefore well within these limits but set to explore the top, middle and bottom of the screw speed range. Compression testing was carried out on the tubes. A clear relationship between extruder speed and the compression load applied to the tube can be seen in Figure 48.

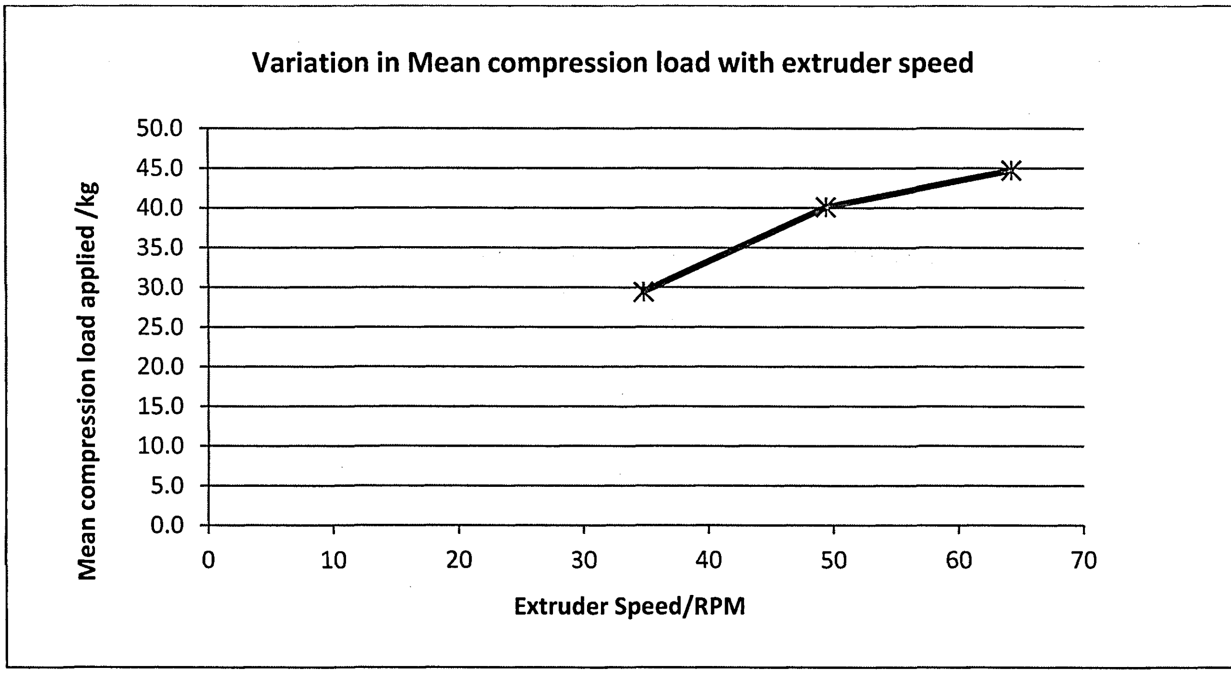


Figure 48 - Variation in compression load versus extruder speed for EPDM/PP tubing

When this compression load versus extruder speed relationship was compared to the pump performance of the tube produced, 36 samples in each batch, see Table 23, clear links can be established. The faster extruder speed results in a consistently higher average tube life.

Tube Batch ID	Mean Life (hrs.)	Extruder Speed (rpm)
#1	323	34
#2	593	49
#3	688	64

Table 23 - Tube life v extruder speed

4.3.3 Mechanical tests and swell tests v_s extruder screw speed

The tube produced is then subjected to swell testing of the material, using the simplified method outlined in chapter 2. The results show a clear relationship between the level of swell displayed by the material and the extruder speed it has been exposed to during the extrusion process, see Figure 49.

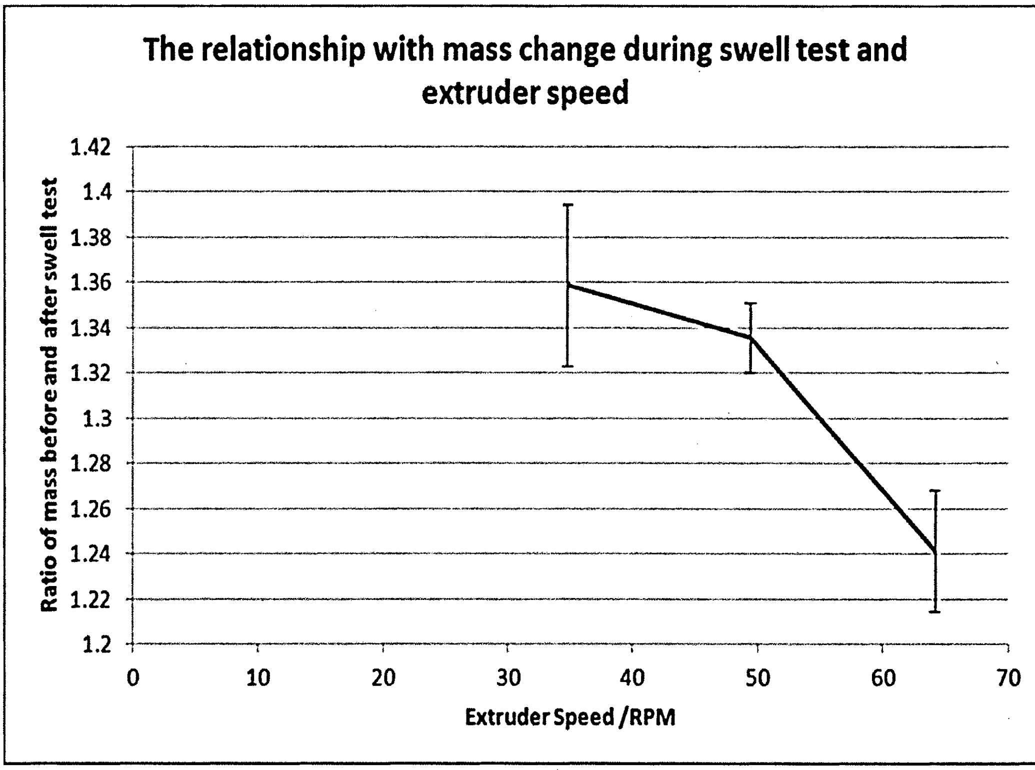


Figure 49 - relationship between level of swell in an EPDM/PP material and extruder speed

The relationship suggests a greater level of crosslinking exists within the EPDM phase of the material run at the fastest extruder speed. With a more effective rubber phase the level of compression load needed to compress the material would be greater, as borne out by the previous results shown in Figure 48.

The variation of compression reaction load with time for each sample is analysed, see Figure 50. It is noted that although the compression load after 60 minutes hold time is greater in the material with the better pump performance, it displays the greatest level of change. The material therefore shows the greatest initial elastic deformation compared to the other two samples tested, before showing the greatest incompressibility.

This behaviour is in line with the observations from the work of others **(66)** who noted that when applying stress to PP/EPDM blends (such as the one used in this study), the polypropylene region acts as a linkage with adjacent rubber domains in the stretching direction. As a result the stress passes through the polypropylene region and the strain is concentrated in the rubber domain. This study therefore suggests that within the rubber domain, the existence of a high crosslink density would result in a more effective rubber network structure, able to adsorb greater levels of strain. It may also be true that the extruder speed has effected a more efficient dispersal of the silica filler within the material, improving the material properties. Work by others **(76)** has shown that dispersion levels of fillers depends on screw rate, melt temperature and screw configuration, as well as the compatibility between the matrix and the filler and the surface free energy of the particle.

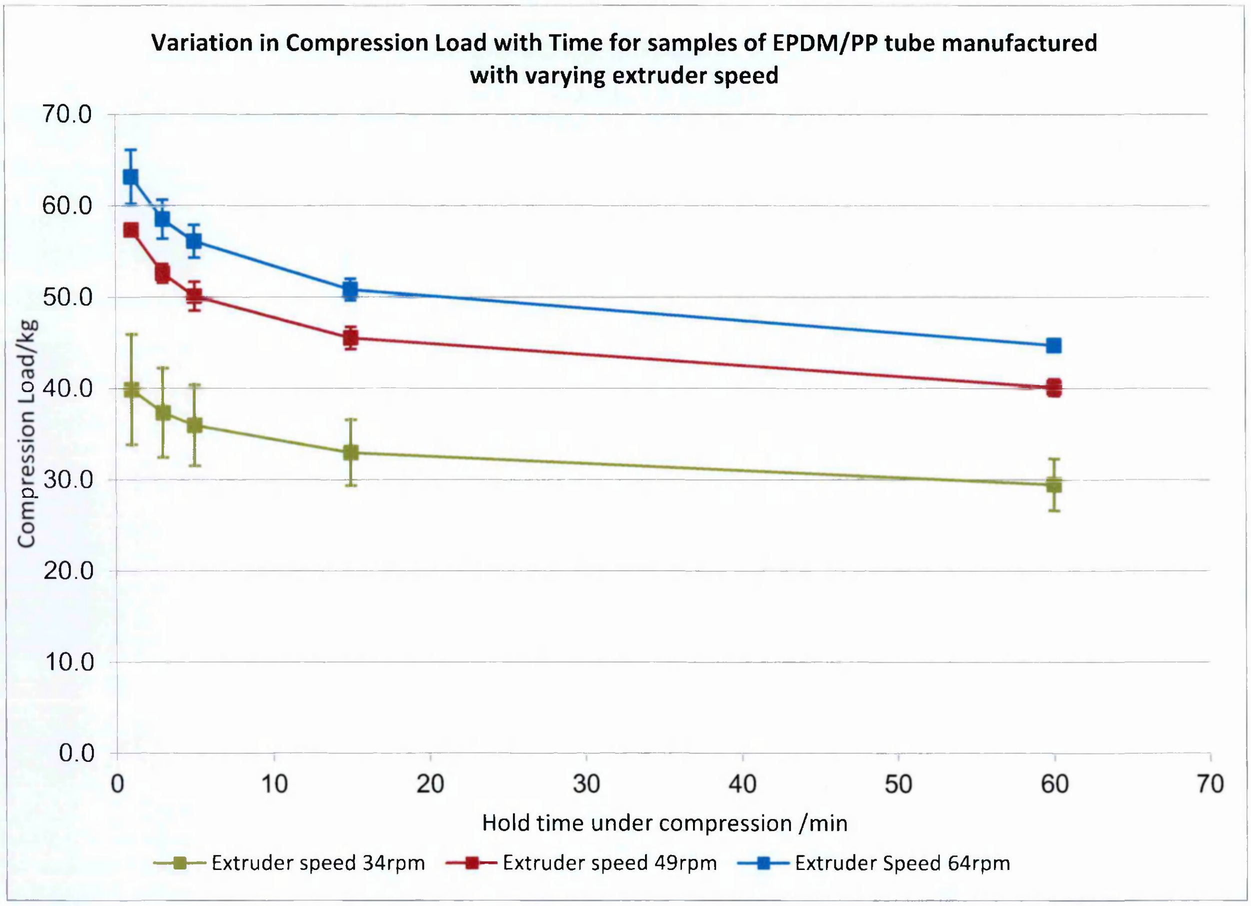


Figure 50 - Variation of compression load with time on samples produced using different extruder speeds

4.3.4 Using extruded test samples

Extruded flat sheet was used to produce standard ISO 37 dog-bone test pieces. These test pieces and tubing are then subjected to tensile testing, outlined in 2.3.5. The results are compared against each other and then against the material certificate values provided by the raw material supplier, which are produced using compression moulded dog-bone test pieces.

Sample ID	Stress at 100% elongation
Extruded dog-bone test sample	5.7 MPa
Tubing test sample	5.7 MPa
Certificate value (compression moulded test piece)	6.1 MPa

Table 24 - Comparison of stress figures at 100% elongation for different test pieces

It can be seen, Table 24, that there is a clear difference between the results for the extruded dog bone and those which use compression moulded dog bones for the certificate value. However, when extruded dog bone results were compared to the results for tensile testing of extruded tube, they are comparable, indeed not just at

100% elongation but throughout the stress-strain curve. It is therefore suggested that the use of the same manufacturing process to produce test pieces and final product is vital if linkages are to be established between standard test schemes and the performance of the material as a peristaltic pump tube.

4.3.5 Conclusions

From the data analysed it is suggested that it is possible to achieve more consistent tube performance using a number of standard test methods on extruded test pieces. It is possible to analyse the influence of extruder speed on the raw material and thereby optimise this part of the manufacturing process to give a more consistent peristaltic pump tube. It has been found that extruded test pieces better indicate the performance of extruded tube than a compression moulded test piece, see Table 24. It has also been found that the use of the tube itself in standard test methods can allow linkages to be drawn more quickly, particularly when using compression tests as an indicator of the optimised extruder speed needed to achieve more consistent performing tube.

It should be noted that this method was only applied to a single tube size and raw material grade. However, what has been illustrated is a type test that can be used as a comparable measure when assessing batches of raw material of the same grade against each other.

It is suggested that the use of a test sequence methodology to build a picture of performance will hold for other sizes of tube and material grades:

1. Tensile test using extruded test pieces to establish extruded data that can be compared to certificate values
2. Compression test versus mix trials versus performance tests to give optimised extruder conditions
3. Compression test versus mix trials versus swell test to confirm phase mechanism and relationship

With a more consistent tube it is easier to assess the influence of the environment on the tube performance and to bring those factors into the prediction strategy and it is these that are discussed in chapter 5.

5 PUMPS AND ENVIRONMENT

5.1 Overview

In order to implement a life prediction algorithm for a peristaltic pump tube, those factors which most influence life of the tube need to be ascertained. It is important for an algorithm to be able to recognise what is normal behaviour for a tube in a pump and what is abnormal behaviour or pre-cursors to failure. However, as noted in section 1.4 the method to build up a predictive algorithm should take account of previous knowledge as part of the building process. Therefore, by analysing the historical data held by a peristaltic pump manufacturer on life, gathered over a number of years, it is possible to gauge whether there are any pertinent patterns that can be used to distinguish which factors warrant further experimental work. The study looks at general distribution first before analysing the results more deeply to look for distribution patterns associated with different environmental factors. It also searches for factors that are not explored in historical data to identify those that need further investigation.

5.2 Historical data – what patterns does it show

5.2.1 General distributions before any consideration of factor influence

Taking data held on tube life and splitting them into the two tubing material groups, one for silicone rubber tubing and one for EPDM/PP TPE tubing, the distribution of life figures can be shown:

5.2.1.1 Silicone Rubber Tubing

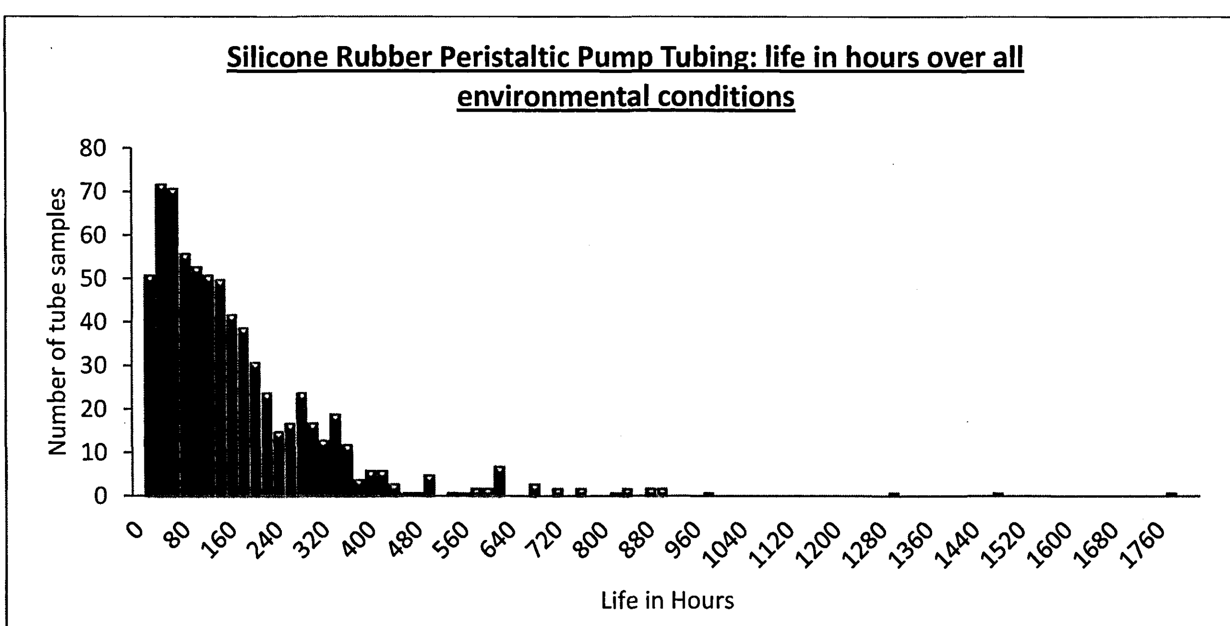


Figure 51 - Silicone rubber tubing - Historical life in all environments tested

Closer examination of the data held showed that the data was taken from experiments run at only ambient temperature, i.e. approximately 21 to 24°C and that all experiments were conducted while pumping water. However the distribution did cover a range of system pressures from around transfer pressure; defined as system pressure between 0 and 0.5 bar, up to 2 bar, a range of different tube sizes, and the experiments utilised a range of pump-heads running at different speeds. The tubing used when gathering these results included both extruded and injection moulded tubing. The raw materials used to produce the extruded tubes were all from one raw material source. The raw materials used to produce the injection moulded tubes were from another raw material source. As can be seen the distribution is a normal skewed distribution for the population which is sized at over 850 samples.

5.2.1.2 EPDM/PP blend Tubing

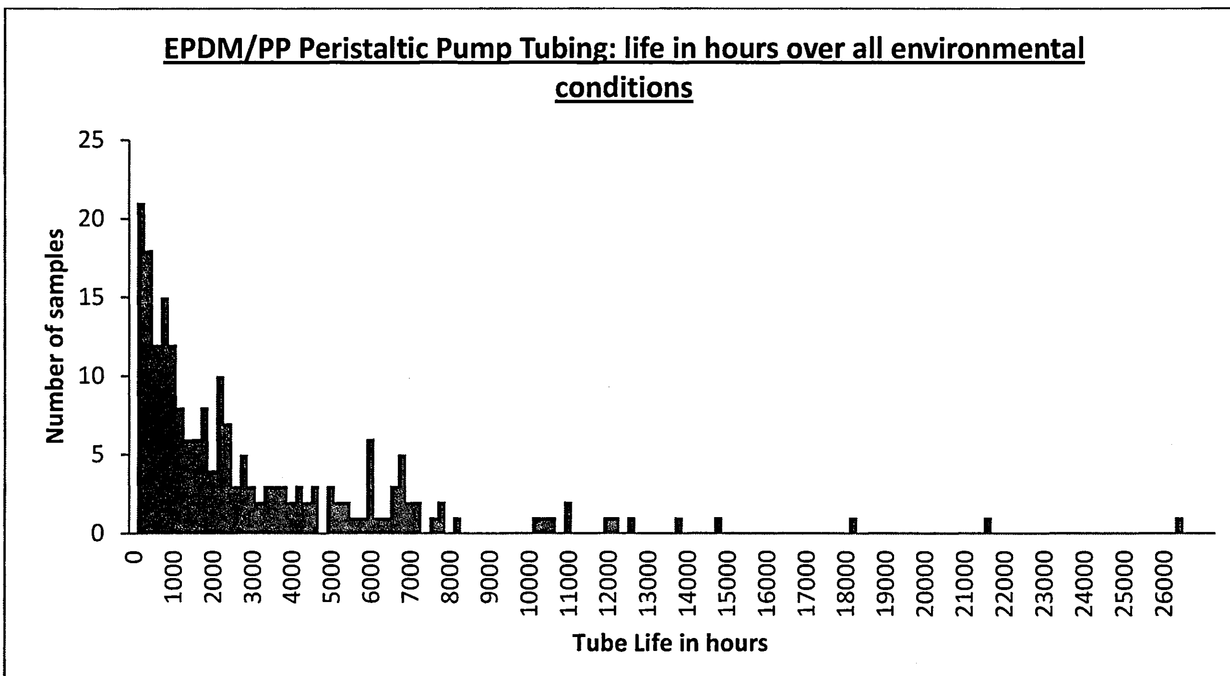


Figure 52 - EPDM/PP Tubing - Historical life in all environments tested

Closer examination of the data held showed that again the data was taken from experiments run at only ambient temperature, i.e. approximately 21 to 24°C and that all experiments were conducted while pumping water.

However the distribution did cover a range of system pressures from around transfer pressure up to 7 bar, a range of different tube sizes and the experiments utilised a range of pump-heads running at different speeds. The tubing used when gathering these results included material grades at a variety of shore A hardness values, from 64 shore A to 87 shore A. The distribution is much wider than for silicone rubber but again shows the same skew pattern, the population size in this case is over 400.

5.2.2 The effect of pressure

When the results for tubes in both materials are separated out into those results at different system pressures, see Figure 53, Figure 54, Figure 55, Figure 56 and Figure 57 below; it becomes clear that as the system pressure increases the life decreases. Tubes under internal pressure can be characterised using a simple hoop stress model:

$$Pd - 2t = Hoop\ Stress$$

(Equation 7)

With increasing internal pressure the hoop stress will rise respectively.

It can be seen that for the silicone tubing in a BW5 pump type the life for a tube working at a system pressure of 2 bars is approximately 30% of the life of a tube working at transfer pressure, see Figure 53. Transfer pressure is defined as system pressures between 0 and 0.5 bar. The population size is over 400 and it can be seen that for this population, at transfer pressure the life can vary considerable, hence the very large standard deviation. Whilst some tubes can last just over 10,000 occlusions, others last over 10,000,000.

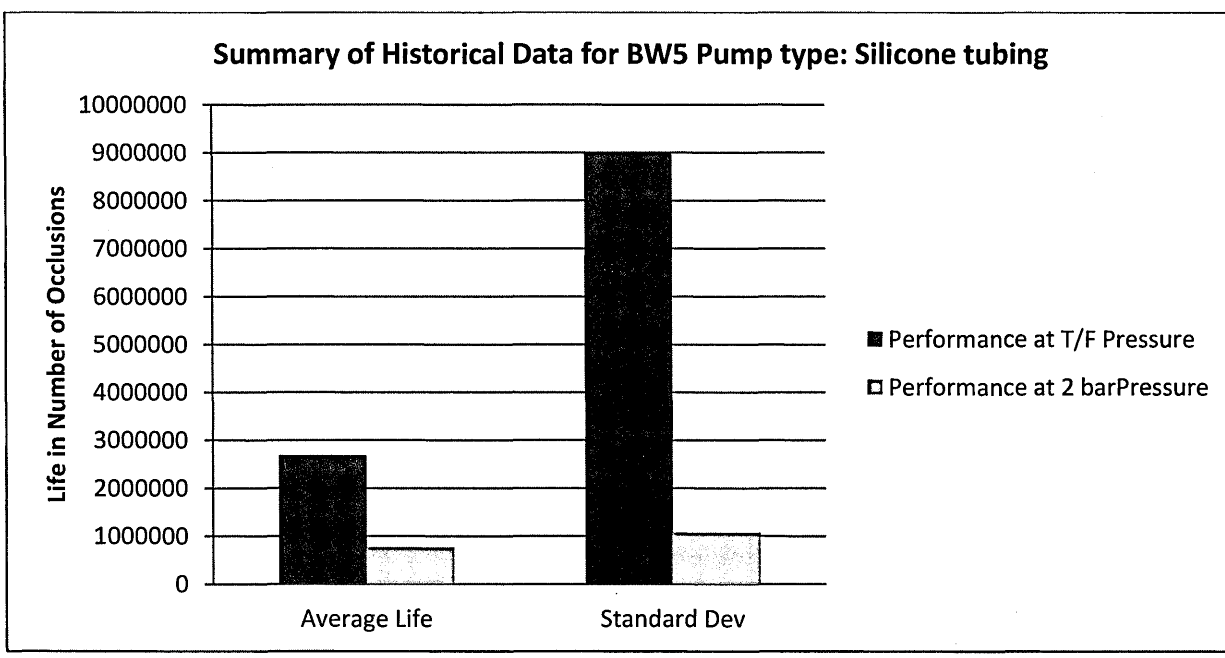
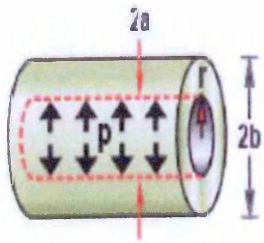


Figure 53 - Historical data: Performance at pressure for Silicone tubing in BW5 Pump

However the relationship between hoop stress levels and pressure is better described for the tubes in this study, defined as thick walled since they a have a radius to wall thickness ration of less than 10, by the following equation:

$$Hoop\ Stress = \frac{pa^2}{r^2}\left(\frac{b^2+r^2}{b^2-a^2}\right)$$

(Equation 8)



Definitions:

p = pressure (N/m²)

a = inner radius (m)

b = outer radius (m)

r = radial coordinate at which measurement is taken (m)

Work by others (11) which models the effect of pumping at pressure on reinforced hoses showed that pumping or system pressure was thought to introduce much larger stresses than could be shown through modelling techniques such as finite element analysis, FEA, and was also therefore thought to contribute significantly to the increases in localised material temperature.

This is borne out by the results for the pump types which take larger sizes of tube which would therefore display larger heat build-up, EW6 and FW7 pump types; see Figure 54 and Figure 55. Population size for the data for each pump type was over 200 samples. Here the difference is even more significant than with the BW5 pump type: the average life of a tube working at 2 bar pressure is approximately 15% of the life seen by tubes working at transfer pressure.

In all cases when working at a higher system pressure the standard deviation of life populations is significantly reduced from working at a transfer system pressure. The distribution of the population narrows considerably, there is not the very wide spread life as seen at transfer pressure.

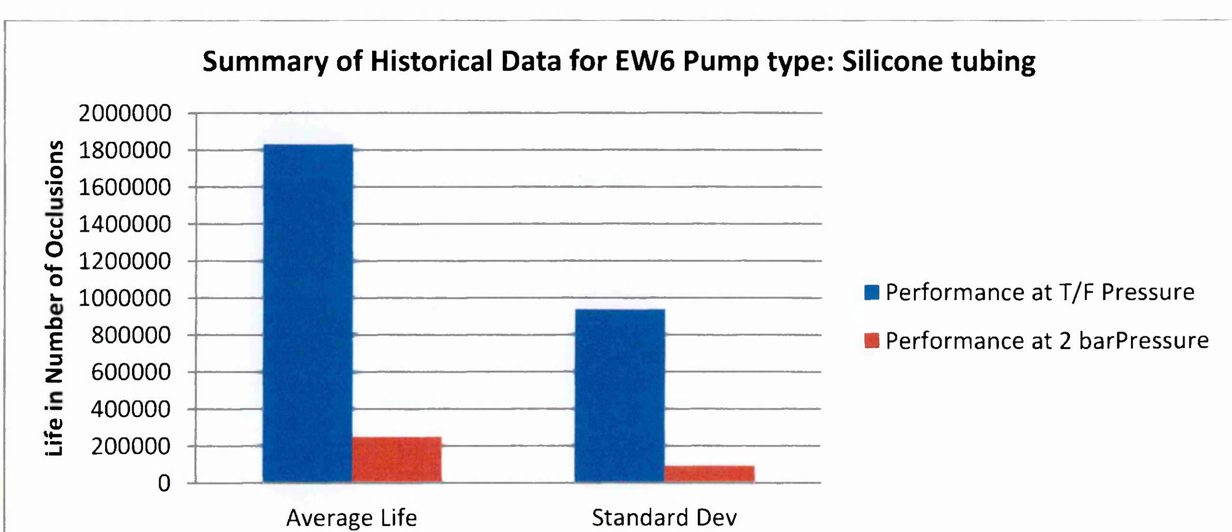


Figure 54 - Historical data: Performance at pressure for Silicone tubing in EW6 Pump

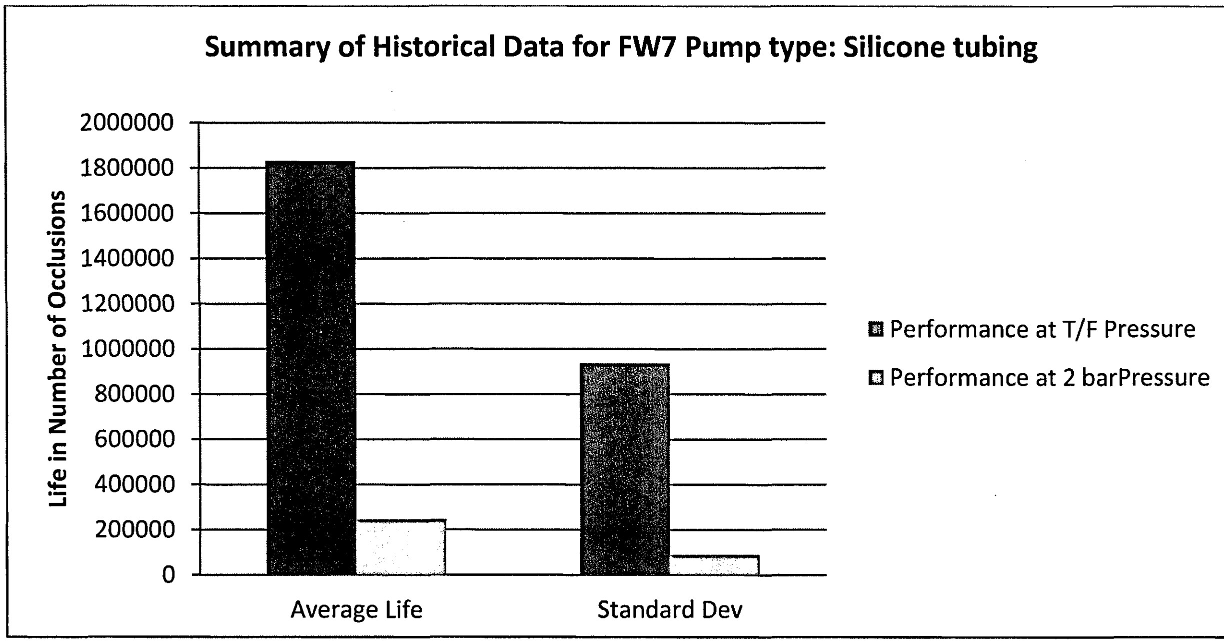


Figure 55 - Historical data: Performance at pressure for silicone tubing in FW7 Pump

The relationship is consistent regardless of tube material. The EPDM/PP blend material shows similar characteristics; see Figure 56 and Figure 57.

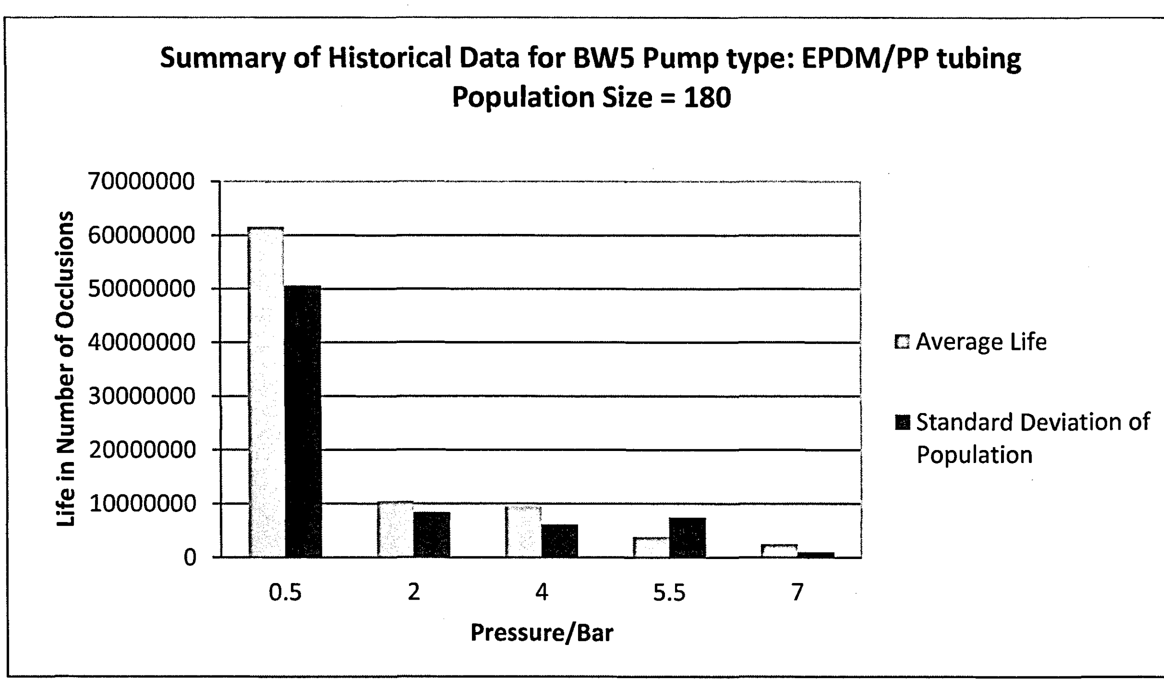


Figure 56 - Historical data: Performance at pressure for EPDM/PP tubing in BW5 Pump

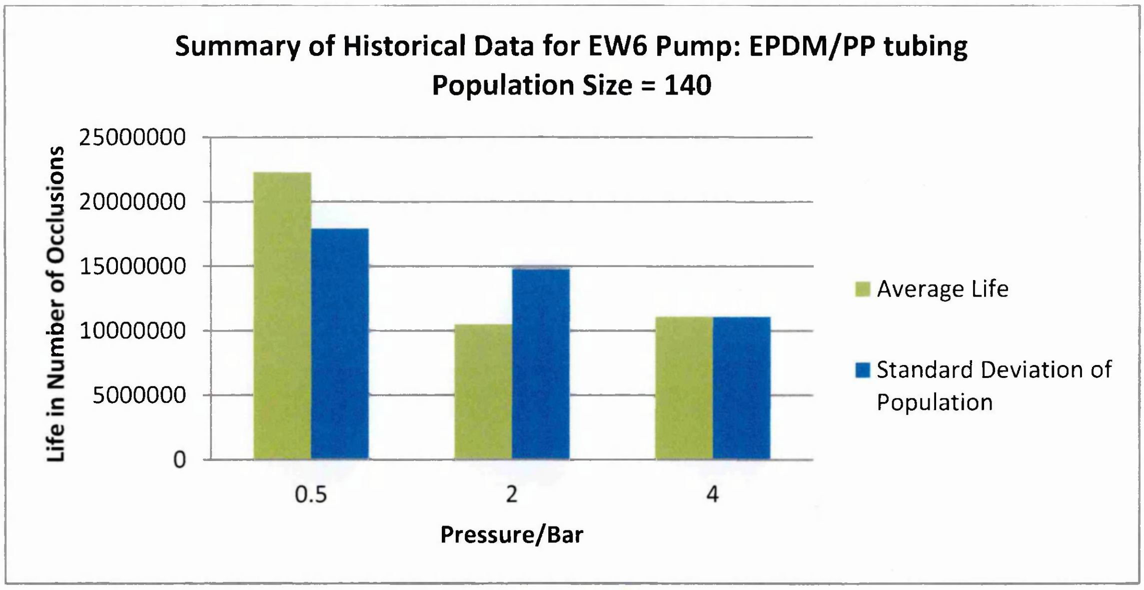


Figure 57 - Historical data: Performance at pressure for EPDM/PP tubing in EW6 Pump

Looking at the results for the tubing produced from the EPDM/PP blends it is noted that the historical data shows for both BW5 and EW6 pump types the difference in life between that at 2 bar pressure and that at 4 bar pressure is fairly small. There seems to be a skew to the data, as one would expect that the greater stress levels the material is exposed to at 4 bars when compared to 2 bars would detrimentally influence the fatigue performance of the tube. This was therefore considered further when performance experiments to look at environment factors were carried out along with the relationship between pressure and life which should therefore be analysed further and was therefore made a factor within the development of the design of experiment (DOE) matrices

5.2.3 The effect of geometric scale – aspect ratio

The aspect ratio of a tube, defined as the relationship between the tube's bore and its wall.

Definitions:
A = Aspect ratio
id = internal diameter
W = wall thickness

$$A = \frac{id}{W}$$

(Equation 9)

It has been shown that 'A' has an effect on how the thermal cycle can influence the performance of a tube, see 4.2.4. When the tube is within a pump the relationship of the aspect ratio to the performance of the tube is more complex. It is known that the aspect ratio can limit the pressure capability of a tube. This is governed both by the hoop stress, see Equation 8, and the tube material properties.

Looking at the distribution of the historical data against the aspect ratio some general linkages are suggested for each tube material. The historical data analysed shows that for silicone tubing the peristaltic pump tubes with an aspect ratio of 5 have a higher average life than tubes with other aspect ratios, see Figure 58. However closer analysis of this historical data shows that tubes sizes which have an aspect ratio of 5 vary considerably in size, from 25.4mm bore x 4.8mm wall to 8.0mm bore x 1.6mm wall. The relationship between aspect ratio and life performance is different for EPDM/PP tubing, for this material those tubes with an aspect ratio of 5 do show higher life, but aspect ratios of 2 and 3 show higher average life, see Figure 59.

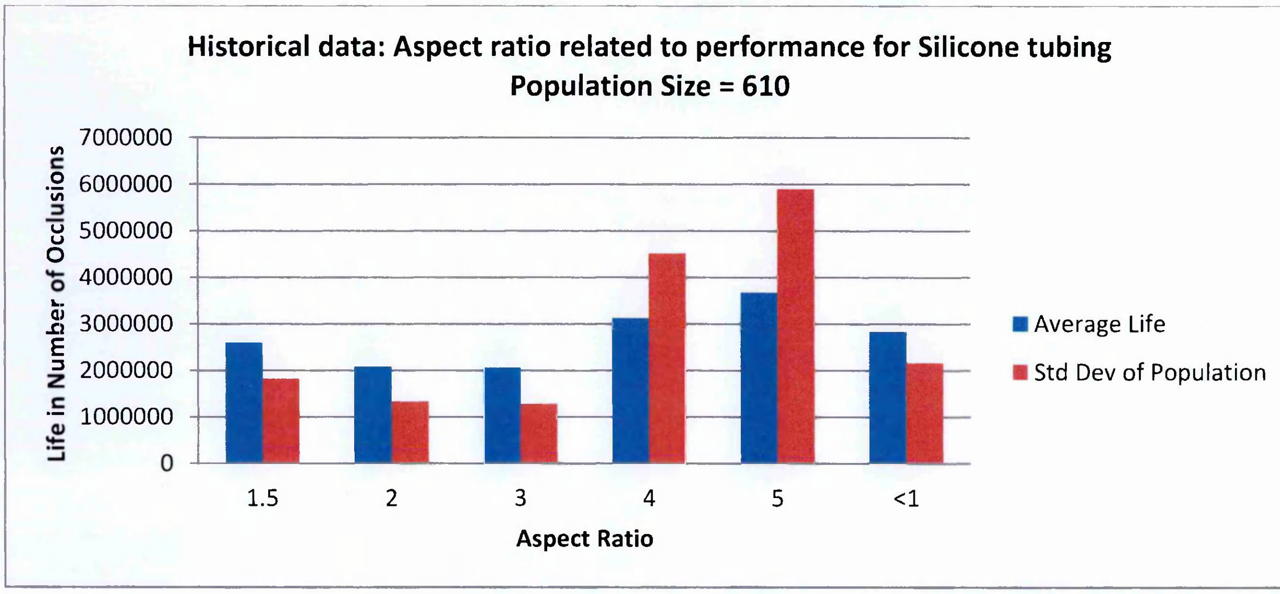


Figure 58 - Historical data: Performance for each aspect ratio for silicone tubing at transfer pressure

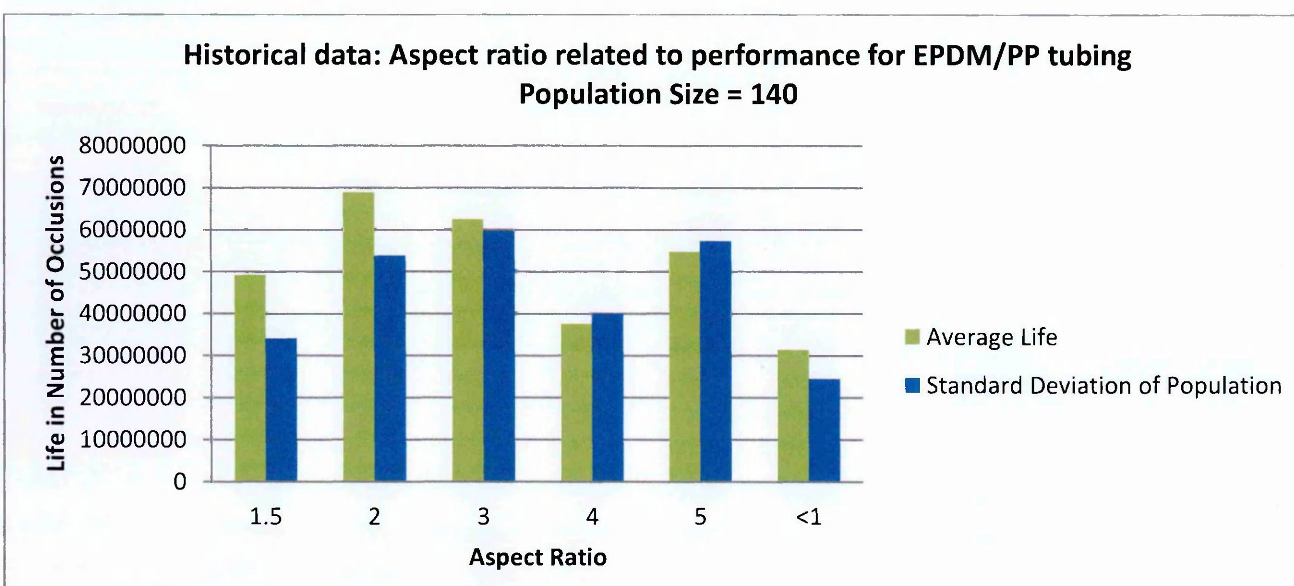


Figure 59 - Historical data: Performance for each aspect ratio of EPDM/PP tubing at transfer pressure

When the detail of the data associated with each wall thickness is brought into the equation the relationship to life differs. The 4.8mm wall thickness tubing shows quite a different relationship across the aspect ratios compared to 1.6mm wall thickness tubing and indeed shows much greater life than the 1.6mm walled tube, as would be expected, with a greater level of resistance to fatigue shown by the larger walled material. However this wall thickness also shows the greatest standard deviation in the life population. The tube with the wall thickness of 4.8mm is used in a different pump to the other 4 sizes, so it is feasible to suggest that this pump contributes to the wide distribution seen for this wall thickness.

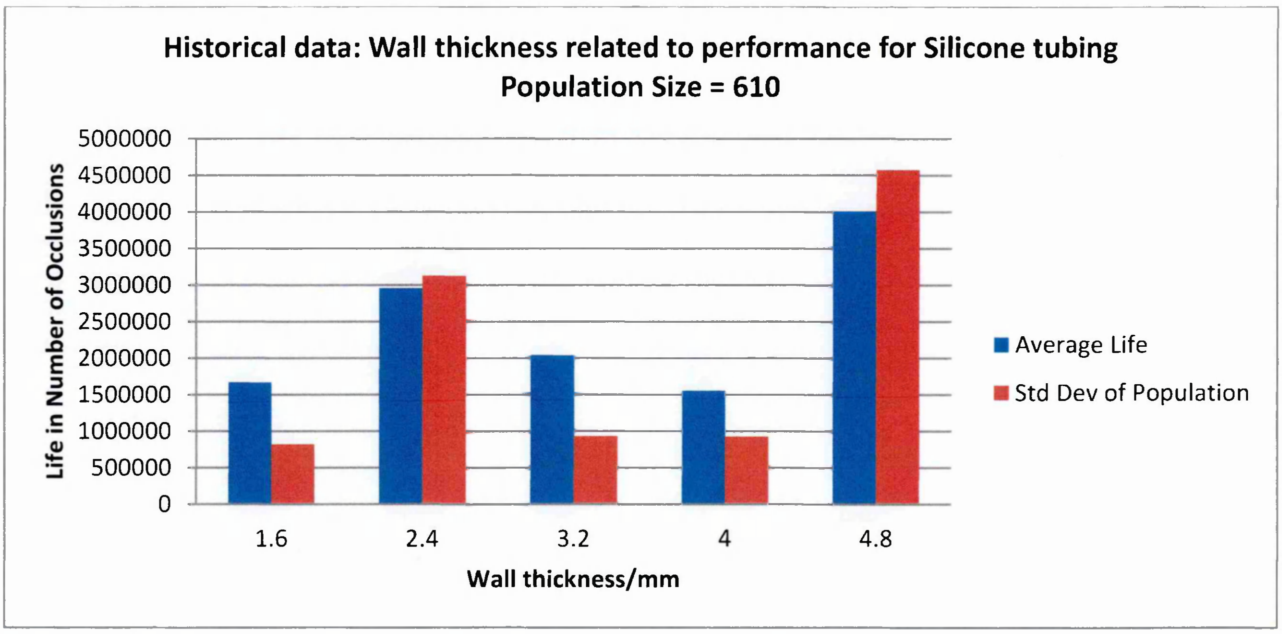


Figure 60 - Historical data: Relationship between wall thickness and life performance for silicone tubing at transfer pressure

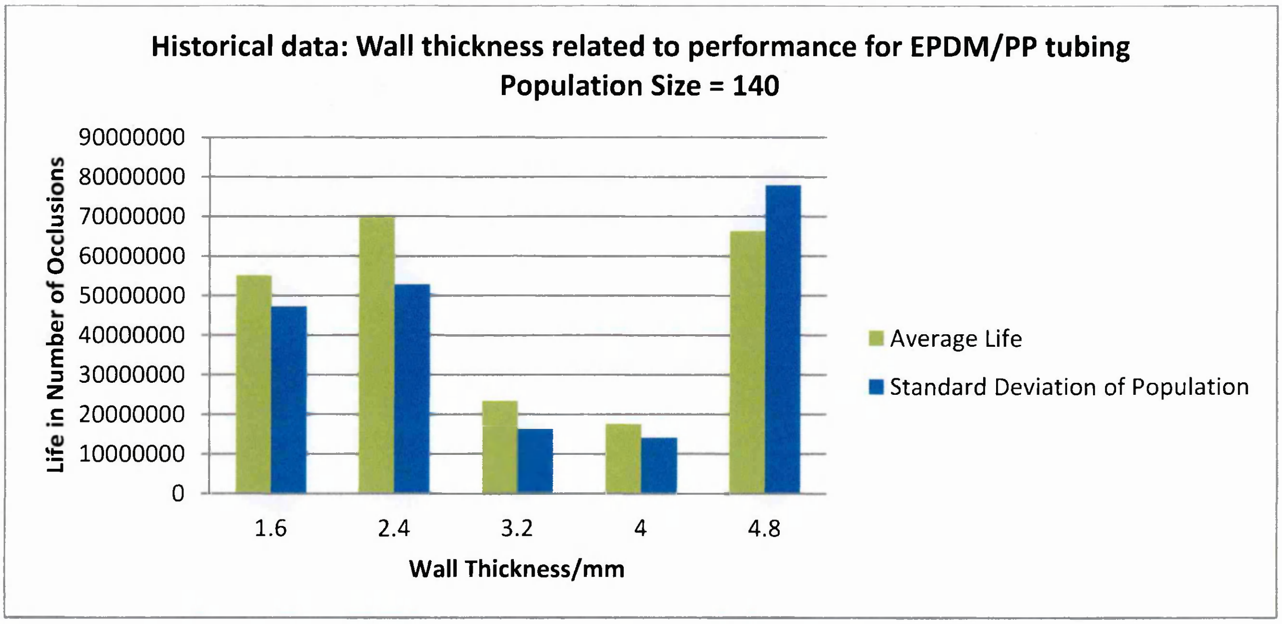


Figure 61 - Historical data: Relationship between wall thickness and performance for EPDM/PP tubing @ transfer pressure

This relationship between aspect ratio, wall thickness and life is material dependent as the analysis of the EPDM/PP blend material results shows.

5.2.4 The effect of using different pump-head configurations

The historical data also suggest that certain pump head configurations may perform better than others. The life results are converted into number of occlusions to remove the influence of the number of rollers in each pump head design from the analysis. When comparing the performance for the same tube size, 8.0mm bore and 1.6mm wall, in silicone rubber material for two different heads, see Table 25, the results from a population of 72 now show that the AW3 head demonstrates a higher average life and lower flow drop performance than BW5 head.

Head ID	Tube Size	Average Life (hrs.)	Average Flow drop
AW3	8mm bore x 1.6mm wall	49	6.6% / 24hr
BW5	8mm bore x 1.6mm wall	78	4.3% / 24hr

Table 25 - Life and flow drop for the same tube size and material on two different heads

The key differences between these two heads as shown in Table 2 Figure 2 are the track material, the track geometry and the lay of the tube within the head. However it should be remembered that the heads tend to be designed specifically for, or optimised for, certain sizes of tube, to achieve a certain range of flow rates.

5.2.5 Using flow profile performance

When the flow profiles for different heads and tubes are analysed it is clear that there are possibilities for using them as part of an analysis engine for a predictive algorithm.

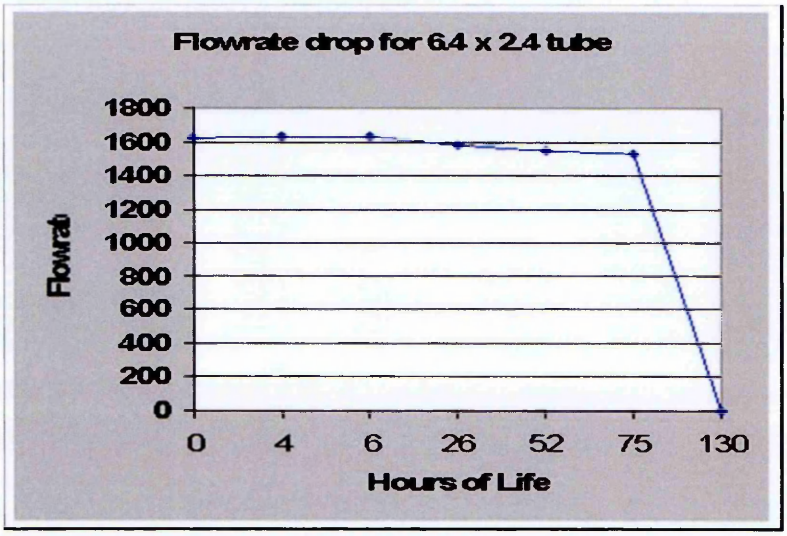


Figure 62 - Flow curve for 6.4mm x 2.4mm silicone tube in a BW5 head

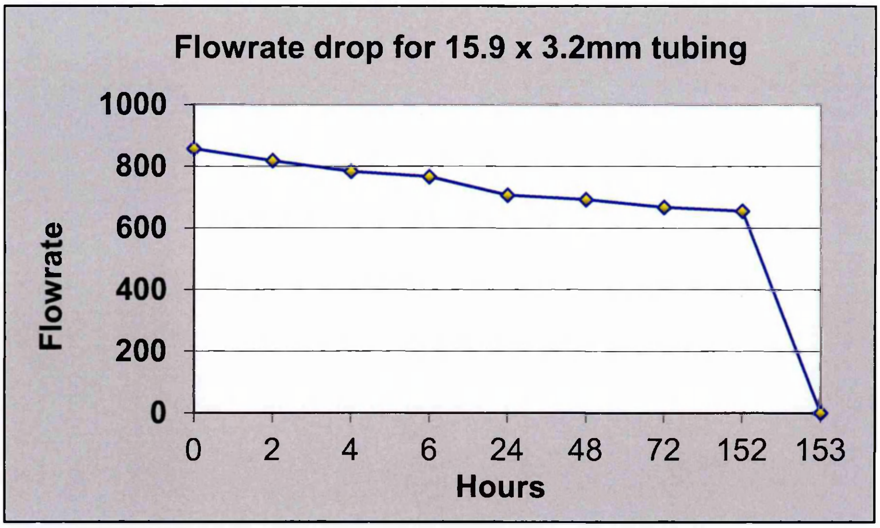


Figure 63 - Flow curve for 15.9mm x 3.2mm silicone tube in a EW6 head

The total flow drop seen over the life of the 15.9mm x 3.2mm size tubing is much greater than for the 6.4mm x 2.4mm size tubing. This relationship holds over a large sample of tubes of the same size in the same head type.

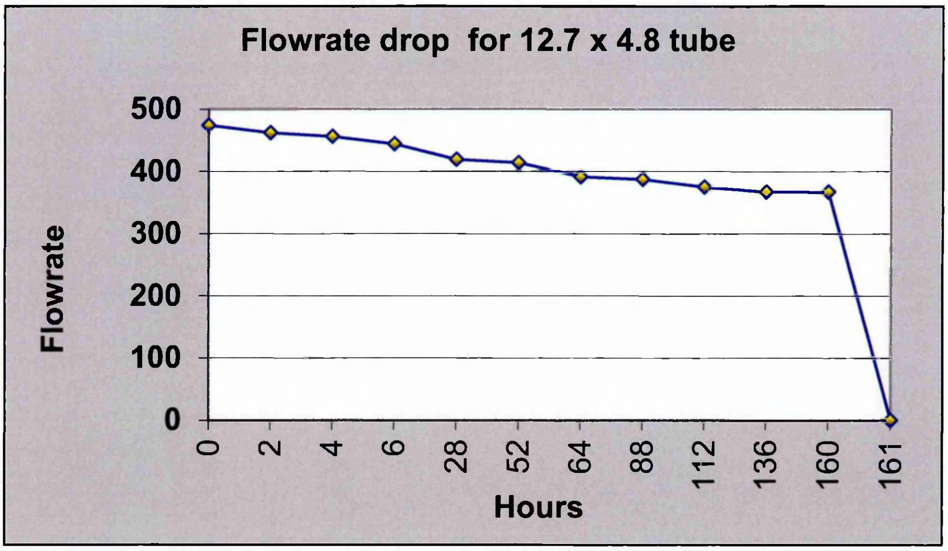


Figure 64 - Flow curve for 12.7mm x 4.8mm silicone tube in a FW7 head

Looking at the flow curve profiles for different tube sizes in different head types it is possible to have a clear pattern of behaviour to follow from which to predict 'normal' behaviours in a set environment.

5.2.6 Conclusions from the historical data: gaps in knowledge

The historical data suggest that the factor that most influences life is pressure and that another factor that should be investigated further is aspect ratio. Work by others (77) (78) into the ageing theory for different polymers highlights the importance of a number of key environmental factors. From this it is understood that one of the most important factors which should be understood further, which has not been explored in the historical work, is the temperature to which the tube is subjected. Not only is the environmental temperature important from a general ageing point of view, but it increases the localised heating within the worked area of the tube. This has the ability to dramatically accelerate thermal degradation processes. The temperature also changes the influence of other factors such as aspect ratio and strain; thicker walled tubes have been found to show a higher temperature rise and greater strains giving rise to greater levels of localised temperature rise (11). This interaction between environmental stresses has been highlighted by others (78) and stresses the importance of looking at interaction between the factors used within DOE matrices.

5.3 Environmental Factors that affect the life of a tube

5.3.1 Silicone tubing

Using the analysis of the historical data a DOE matrix can be designed to explore factors affecting the life of tubes, starting with silicone rubber, being a material which can explored more quickly than an EPDM/PP blend material due to its shorter life span. The experiment set uses a matrix, shown in Figure 65, which allows 4 factors to be explored to 3 differing levels within a 9 experiment set. It can be seen that the factors the experiment looks at changing are temperature, wall thickness, aspect ratio and speed. The experiments either run at a temperature of 4˚C, 23˚C or 40˚C, the tubes chosen either have a wall thickness of 2.4mm or 1.6mm, with the pump running at 100, 220 or 300rpm. The aspect ratio determines which size tube will be chosen. For example an aspect ratio of <1 will result in the experiment utilising a tube size of 0.8mm bore with a 1.6mm or 2.4mm wall. The tube size choice is a fixed factor, factor types are discussed in section 2.3.3.

Factor		**1** **A** **Temp** **˚C**		**2** **B** **Wall Thickness** **mm**		**3** **C** **Aspect Ratio**		**4** **D** **Speed** **rpm**	
Level	**3**	40		2.4		4(5)		300	
	2	23		2.4		3		220	**Analysis**

Run	1	4		1.6		<1		100	No
1	1	4	2	2.4	2	3	2	220	2
2	1	4	3	2.4	3	4	3	300	3
3	2	23	2	2.4	3	4	1	100	5
4	3	40	2	2.4	1	<1	3	300	8
5	3	40	3	2.4	2	3	1	100	9
6	1	4	1	1.6	1	<1	1	100	1
7	2	23	3	2.4	1	<1	2	220	6
8	2	23	1	1.6	2	3	3	300	4
9	3	40	1	1.6	3	4	2	220	7

Figure 65 - DOE Matrix of experiments for silicone rubber

5.3.1.1 Temperature and silicone rubber

Silicone rubber tubing in a single raw material produced using the same extrusion process is used to carry out the experiment set outlined by the matrix above. The results from a population of 162, Figure 66, show that the temperature the silicone rubber is subjected to whilst in the pump affects life dramatically.

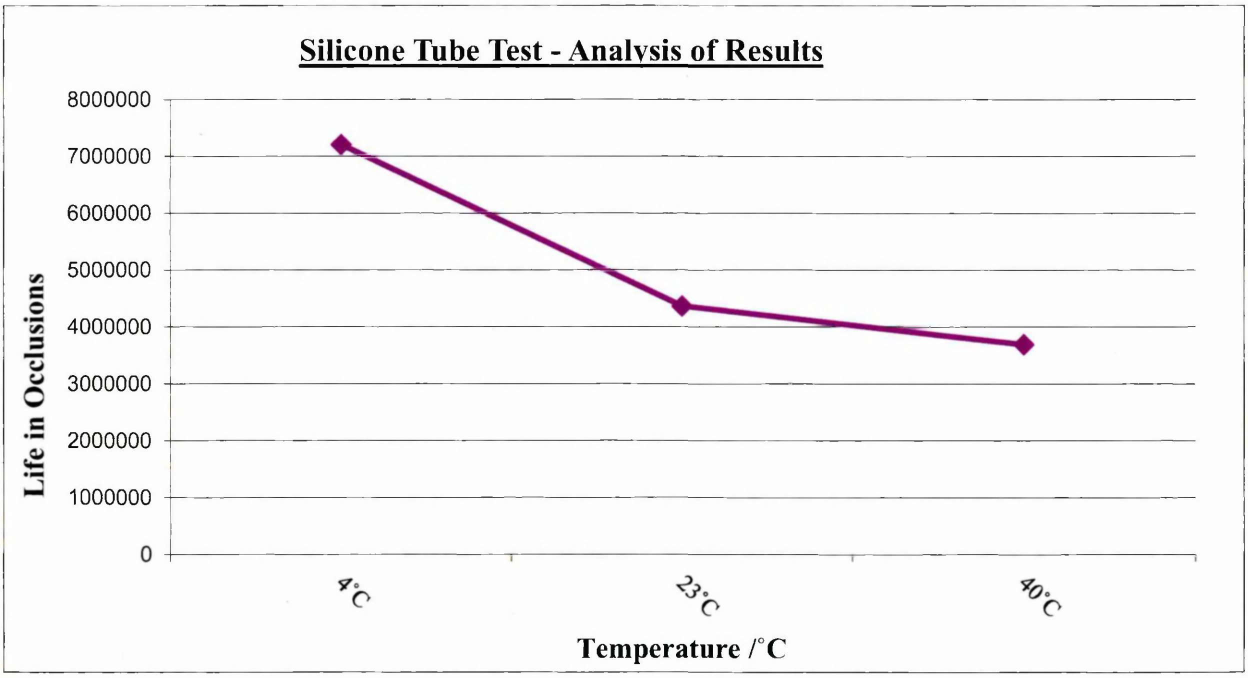


Figure 66 - Effect of temperature on the life of silicone tubes when pumping water

At temperatures of just 40°C the life of silicone rubber can be reduced by over 15 % from the life experienced at room temperature, i.e. circa 23°C; however, at running environments of 4°C the life of the tube increases by over 65% of that shown at room temperature. This experiment was then repeated with tubes extruded from two further silicone raw materials to see if the relationship remains consistent, see Figure 67.

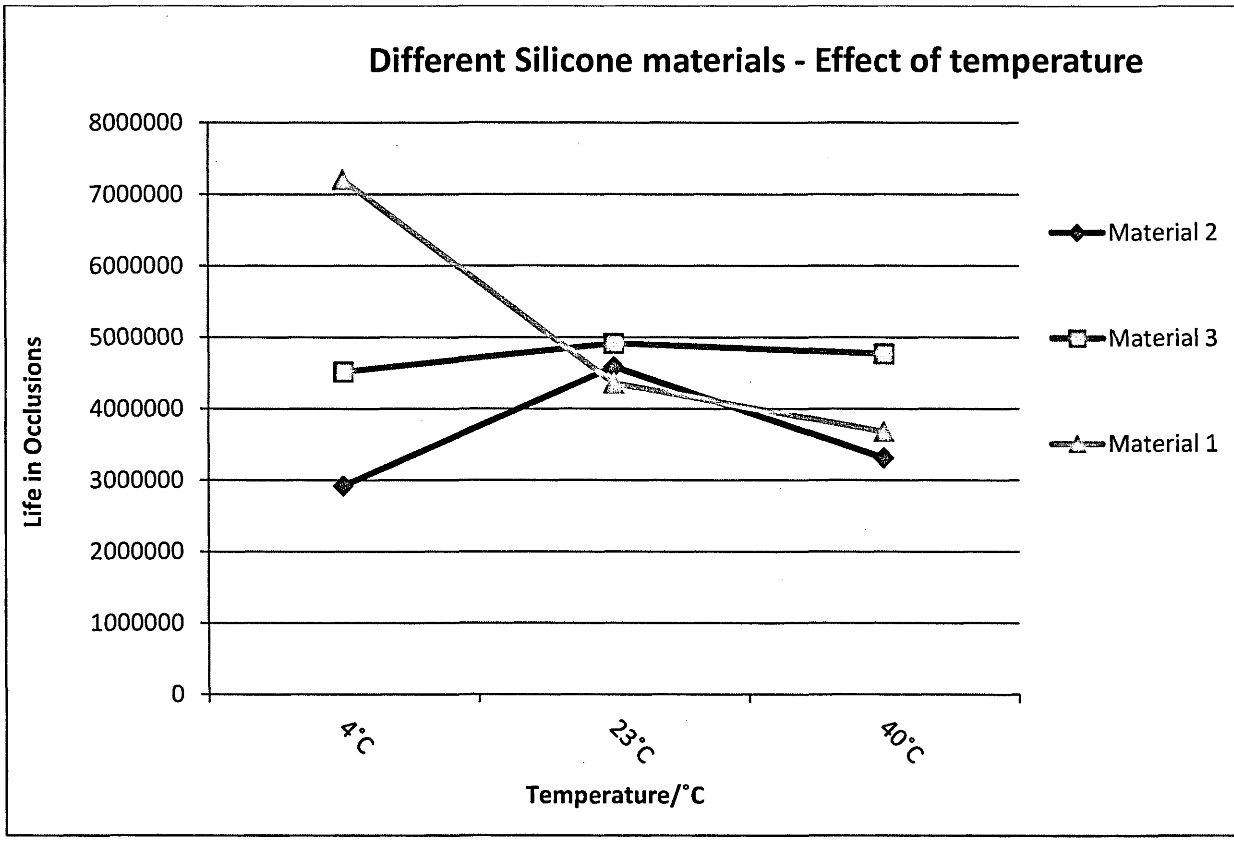


Figure 67 - Effects of temperature on the life of different silicone materials when pumping water

The relationship between temperature and tube life for silicone rubber has been shown to be dependent on the raw material used. The association of high temperature with lower life is true for all silicones tested. However the association of greater life with lower temperatures does not remain constant.

Degradation of polymers has been found to be influenced by the potential reactivity between the polymer and its degradation products, which can influence in either a positive or a negative sense the stability of the product (78).

Noting the differences in materials identified by the key indicators and performance in chapter 3 it is apparent that the network structure of silicone rubbers can vary considerably, it is therefore necessary for predictive purposes before a life model can be applied to ascertain which category of relationship a silicone rubber falls into:

- Low temperature = higher life than ambient
- Low temperature = lower life than ambient

5.3.1.2 *Pressure and silicone rubber*

The relationship between system pressure and tube life performance can be analysed by comparing the results for a number of tube sizes, run under the same environmental conditions with only system pressure being altered between 2 bar and transfer pressure.

It can be seen, see Table 26, that for the tubes tested the average life at 2 bar is always about 0.2 of the average life at transfer pressure, regardless of head type. There is a slight trend as the aspect ratio increases for the ratio between life at transfer and at life at 2 bar to also increase, but not so much that the same relationship could not be used within an estimation model from which prediction could be built.

Tube Size	Aspect Ratio	Head Type ID	Pressure	Life (occlusion)
9.6 x 4.8	2	FW7	0	2,208,000
9.6 x 4.8	2	FW7	2	384,000
9.6 x 3.2	2.5	EW6	0	1,780,800
9.6 x 3.2	2.5	EW6	2	373,650
6 x 2.4	3	BW5	0	1,504,800
6 x 2.4	3	BW5	2	324,000
15.9 x 3.2	4.9	EW6	0	2,130,600
15.9 x 3.2	4.9	EW6	2	469,050
25.4 x 4.8	5.2	FW7	0	5,568,000
25.4 x 4.8	5.2	FW7	2	1,296,000

Table 26 - Comparing tube life at different pressure

5.3.1.3 *Aspect ratio and silicone rubber*

The aspect ratio is explored using just one head type for three different silicones see Figure 68. Two of the silicones show very similar relationships between aspect ratio and life. The other material shows a different relationship. However when we consider that these same three materials showed different relationships between life and temperature it seems clear that there are quite different network structures which are likely to perform differently at different geometric ratios. What this shows is that a preliminary study of aspect ratio versus life should be done before a silicone material is added into a prediction mechanism to establish which relationship should be applied.

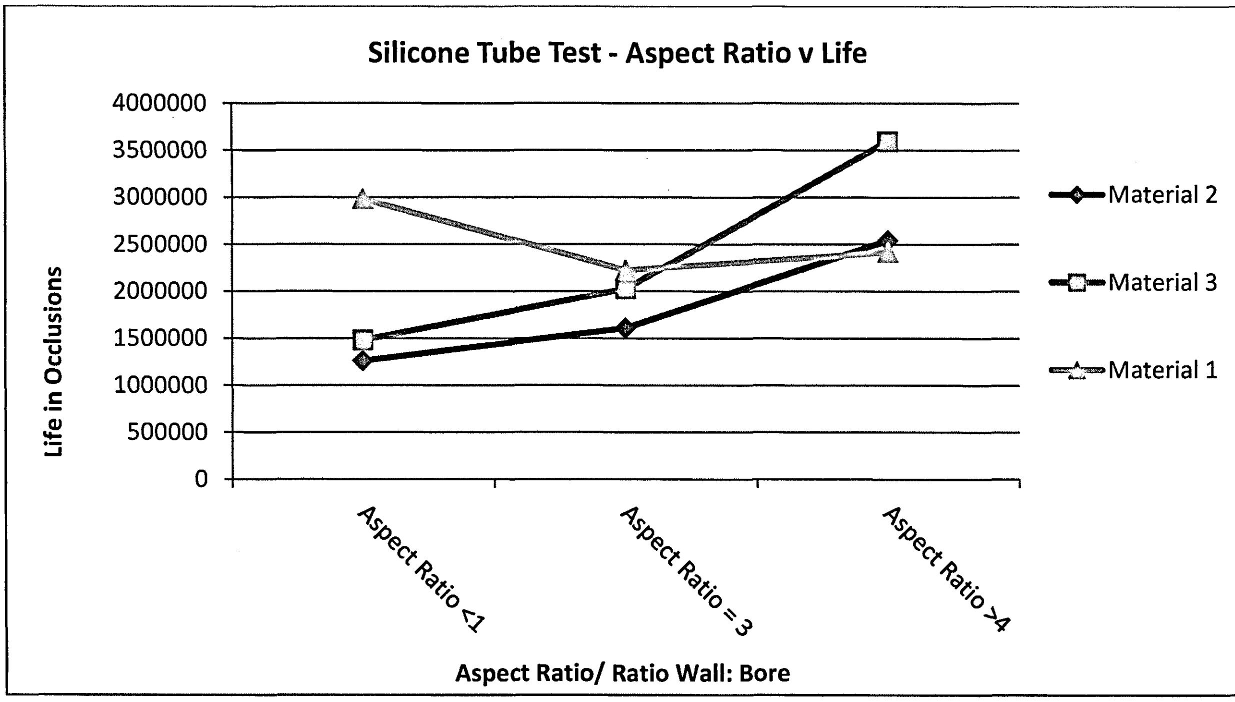


Figure 68 - Relationship between aspect ratio and life

5.3.1.4 Speed and silicone rubber

When the speed that a tube is run at is compared against the life that it is achieved a clear relationship can be seen, see Figure 69.

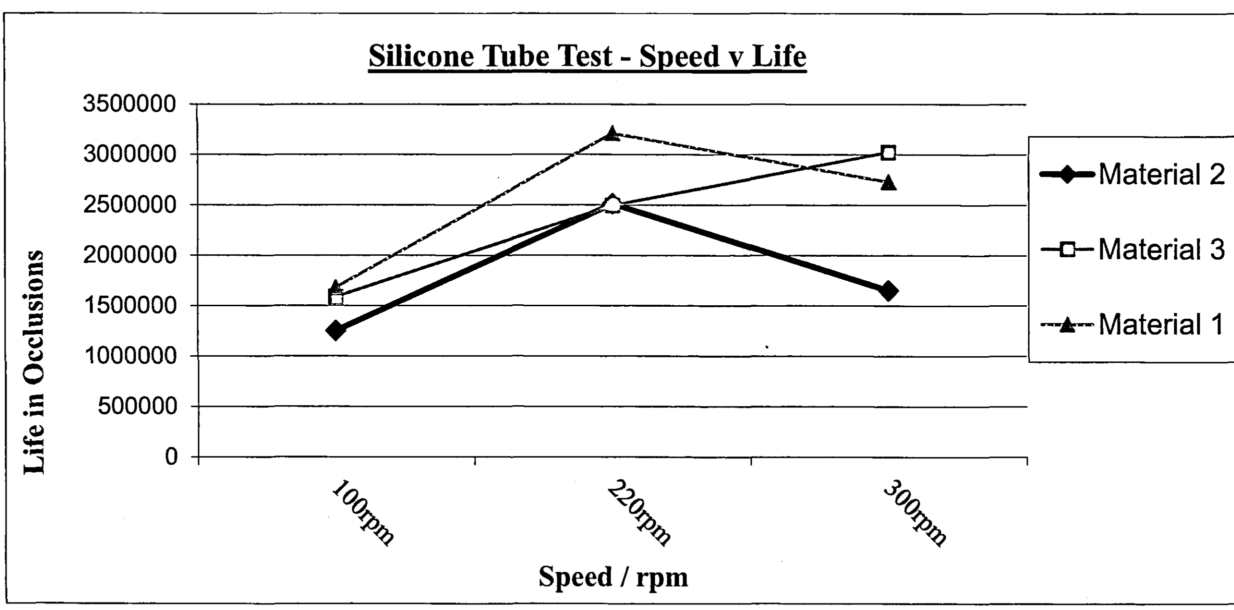


Figure 69 - Relationship between speed and life for silicone tubes

The relationship is surprising: it was expected that the lower the speed the higher the life would be, because of the lower number of fatigue cycles imposed on the tube material. However as this is not the case in the experiments carried out, this may mean that the interactions at lower rotor speeds are more complex in terms of the stress applied to the tube and the frictional interactions that occur. The algorithm design for prediction should therefore take this into account.

5.3.1.5 Interaction between factors

It is important to take into account within any prediction mechanism that there is likely to be interaction between factors, as has been noted from previous studies and by this work. For example it has been shown that pressure and temperature can interact, with high pressures resulting in localised heat build-up within the material and high temperature can result in material changes which influence the effect of system pressure on the tube. Speed and temperature can interact. This relationship is not simple as the relationship between speed and life shown previously in Figure 69. When the interaction between the factors is shown, see Figure 70, it can be seen that the relationship is not straight forward.

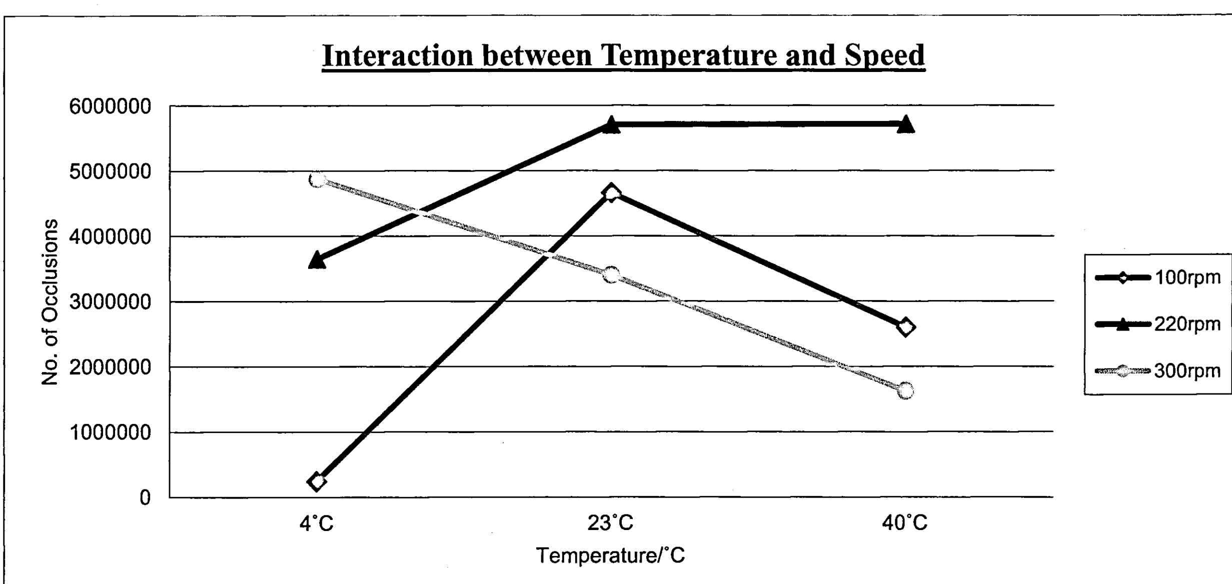


Figure 70 - Interaction between temperature and speed and the effect on tube performance for a silicone rubber

Further interactions are shown within the appendices 18, 19 and 20 shown in 9.17, 9.18 and 9.19.

5.3.1.6 Other environmental factors to consider for silicone rubber

Although not covered by this study it is noted that the chemical composition of the medium being pumped needs to be considered within the algorithm. It has been seen that it can detrimentally effect the tube and contribute to early failure through modes such as environmental stress corrosion cracking, see Figure 33, page 55, and a major reason a user selects a certain tube material will be due to the chemical compatibility between the tube material and the medium they are pumping, see 9.20 Appendix 20: Chemical Compatibility Tables for

Silicone rubber and the thermoplastic rubber EPDM/PP blend It is possible that simple chemical grouping classes can be used initially in an estimation model. These can then be improved upon as more data is gathered by the pump to allow the algorithm to 'learn' how the tube is responding to the chemical it is pumping. This could be through entry of life achieved for a larger sample of tubes in a process, which the algorithm can integrate into its database of knowledge. The viscosity of the fluid being pumped will also have an effect on life, decreasing the flow and changing the stresses seen within the tube wall. It should therefore also be considered more fully for an algorithm to be able to reflect real applications as fully as possible.

5.3.2 EPDM/PP tubing

The approach to find out how tube made from EPDM/PP material performs under different environmental conditions differs to that used for silicone tubing. From the historical data it can be seen that EPDM/PP tubing lasts a good deal longer than silicone tubing, therefore in order to gain enough understanding of relationships between the tube and its environment the experimental approach must differ slightly. Factors are used together to reduce life: for example a high system pressure is used to bring the average life expectancy down, the temperature of the environment is then varied between high, ambient and low to see how this influences life. The experiment set works with a single head type and single material variant to reduce the influence of other variables on the results.

5.3.2.1 Temperature and EPDM/PP materials

Using a high system pressure to reduce average life, the effect of the environment temperature was analysed. EPDM/PP tubes of the same size, 6.4mm bore x 3.2mm wall thickness were run on the same CWM head type, pumping water at 125rpm. The environment that the pumps are run was set to 40, 22 or 4˚C and the life of the tube was recorded. The results from a population of 48, see Table 27, show that the life at 40˚C just over one third the life seen at 22˚C ambient, whilst at 4˚C the life is reduced by 33%.

Environmental Temperature	Average Life Achieved
40˚C	500 hours
23˚C	1600 hours
4˚C	1000 hours

Table 27 - The effect of temperature on the life of EPDM/PP blends when run at 7 bar

In earlier work by the author, the local temperature on the surface of an EPDM/PP tube running at transfer system pressure in a pump was found to reach 135˚C when running at 23˚C. It was found from an earlier study

(11) that higher system pressures also cause internal temperature build up, therefore the 40˚C environmental temperature is likely to cause localised hot spots on the tube at temperature in excess of 135˚C. The melt temperature of the EPDM/PP blend is 130 – 168˚C; therefore degradation will be accelerated at 40˚C as shown by the reduction in life. The reduction in polymer properties at the higher temperature has also been highlighted by a number of studies into polymer degradation through thermal degradation (80) (78).

5.3.2.2 Pressure and EPDM/PP materials

The effect of pressure on the blend was explored through a series of tests. Utilising the CWM head type, a single tube size, 6.4 x 3.2mm and a single pump speed, 125rpm, a population of 60 tubes were run at 5 different system pressures.

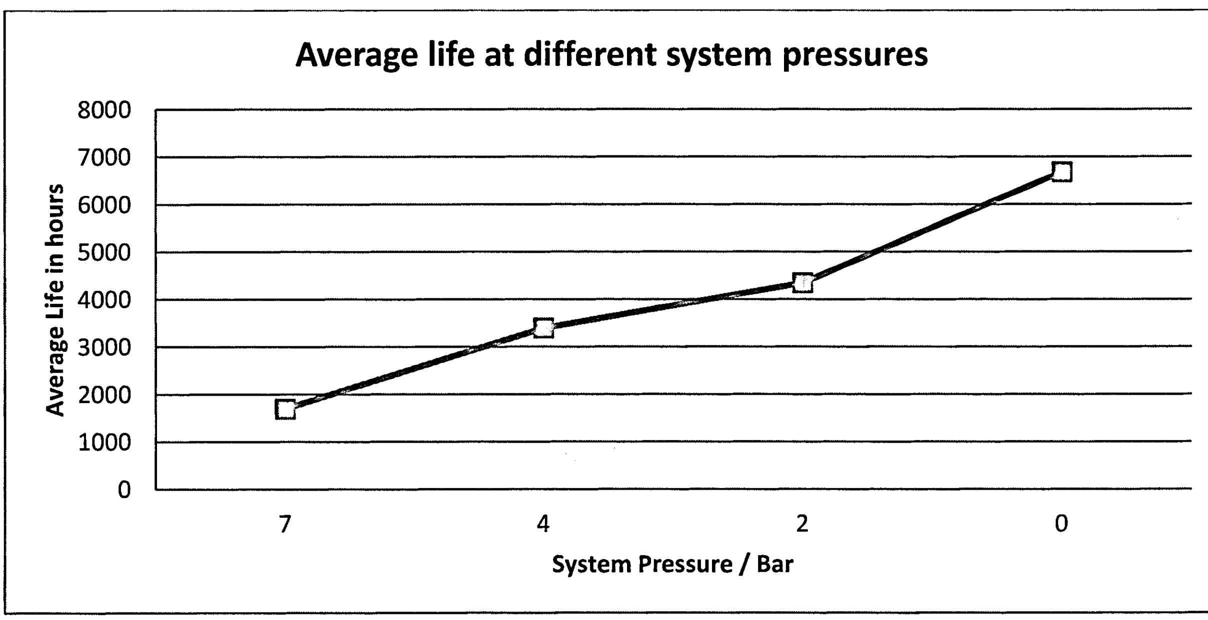


Figure 71 - Average life at different system pressure for a single head type

The relationship between system pressure and average life is linear in nature as shown in Figure 71, however once system pressure reaches 10 bars the relationship is not maintained. The life at this system pressure falls to less than 24 hours. At 10 bar the hoop stress exerted on the tube is 20 bar, although the tensile strength at break for the raw material is given as 155.8 bar it has been found that the high system pressure produces not only the hoop stress exerted on the tube but an increase in localised heat build-up. This combination of high internal pressure and localised heat build-up, along with the shear and frictional forces being exerted on the tube during pumping mean that the tube material performance tails off dramatically at 10 bar, and only seems to perform reliably to 7 bar.

5.3.2.3 Speed and EPDM/PP materials

When the relationship between rotor speed and tube life is examined the relationship pattern shows similar trends to that seen with silicone materials. An 87 Shore A EPDM/PP material blend was used to produce tubing. A 7 bar system pressure was again used to accelerate the experimental results, the temperature in which the experiments are run was kept at circa 23° C. Using the CWM head type a population of 48 tubes pumping water were tested at 3 speeds, 125rpm, 50rpm, and 5 rpm, see Table 28.

Test Speed in rpm	Average Life in hours	Average Life in No of occlusions
125	1691	1,268,2500
50	2983	8,949,000
5	8339	2,501,700

Table 28 - Life v speed for EPDM/PP blends material

When the average life in hours is viewed the low speed running seems to offer considerably longer life than at high speed running. However when the average number of occlusions that the tube has sustained the high speed running is considered, the high speed tube withstands over 5 times the number of fatigue cycles as the low speed tube and 1.4 times that of mid speed running. A prediction algorithm must take this relationship into account when calculating the life a tube is expected to last.

5.3.2.4 Other environmental factors to consider for EPDM/PP materials

As with the silicone rubber material, there are a number of other environmental factors that should be considered within the development of an algorithm. One of these is whether the tube has been subjected to any sterilisation, particularly gamma irradiation: it is known through the work of others that irradiation plays an important role in the degradation of TPE's, particularly EPDM/PP blends, where it can lead to chain scission of the PP phase and changes in the cross-linking in the EPDM phase (80). These effects along with the interaction seen between internal pressure, the related stresses and localised temperature build up mean that the influence of environmental factors needs careful consideration within the algorithm mechanism. This is where the entry of life data by the pump user can help the pump 'learn' what additional influences may be affecting the tube performance on top of those already taken into account. The additional factors discussed for silicone rubber

5.4 Sensor usage – the inputs to the algorithm

For an algorithm to work, it is necessary that it can 'see' the environment in which it is working. In the basic outline algorithm design shown in Figure 22, these inputs are shown as sensors. From the historical data analysed and the experimental results shown in chapter 5 it becomes clear that the sensor inputs should cover the following variables in order to analyse the factors that will influence the performance of a tube within the pump:

1. System Pressure – Discharge & Suction
2. Temperature
3. Flow
4. Speed

It is possible to use sensor systems already available within industry to input this data into an algorithm. It is also possible to use the intelligence within the pump to discern a number of these variables. The intelligence within the pump is the micro-processors and embedded software which is used to control it. Thus for example the one of the factors identified, speed, is constantly being monitored by the pump's control software and could be monitored by the algorithm suggested. The other sensors could also be integrated as inputs into the existing pump processor and thus be processed using this on-board intelligence.

6 DEVELOPING LINKS IN THE LIFE CYCLE - PATTERNS IN BEHAVIOUR

6.1 Overview

The final part of the life cycle shown in Figure 23, page 34, and its methodology shown in Figure 24, page 35, is to establish the patterns of behaviour for the tube types analysed. This involves establishing links between the three other parts of the tube life cycle and the tube itself. It also involves looking at potential sensor sources for pre-cursors to failure which may be used in a prediction mechanism.

This section aims to draw together the work done in the first three areas of the life cycle, highlighting key linkages and patterns in behaviour. It considers tubes from two material groups separately and builds up a picture of how they perform in different pump systems and environments. It looks at symptoms of failure within this system and how they can be monitored through sensors which can be used to both feed an analysis engine and check historical data against, to allow a 'learning' aspect to be introduced.

6.2 System 1 – the Silicone tube life cycle

6.2.1 Raw Material

Raw material data supplied by the material supplier can be used to establish basic links to historical performance data. More detailed tube performance data for material which displays the following properties has been gathered:

- Hardness – 55 to 65 Shore A
- Ultimate Tensile Strength – 11 to 11.2 N/mm²
- Elongation at break – 500 to 800%

If silicone material which displays raw material data within these bands is extruded into tubes using the same dimensional tolerances as used for the historical tubes, then broadly comparable tube life results can be established. The data suggests that the material which shows an elongation at break towards the top end of the tolerance band will display longer life. However if key material indicator tests are used, then the link between raw material data and performance can be strengthened by better characterisation.

1. Silicone materials that show lower levels of total extractable material after extraction with n-hexane suggest a more fully bonded network structure which will perform better within a pump.

2. Silicone materials that show lower levels of swell in toluene suggest higher levels of crosslinking within the bulk and also higher levels of silica filler, this network structure is more resilient and performs better within a pump.

3. Silicone materials with low levels of the volatile compound OMCS tend to display better performance as a pump tube. Better performance is also linked to the material showing OMCS levels which remain unchanged or decrease when the material is subjected to irradiation and which decrease after thermal ageing at 123˚C.

6.2.2 Manufacturing Process

The influence of the manufacturing process on how well a silicone material will perform as a peristaltic pump tube is explored. Analysis of limited historical data for injection moulded silicone tubing $v_{s.}$ extruded tubing has shown that injection moulded silicone tubes can display considerably greater life than extruded tubing. However, it is when one process, extrusion, is explored in some detail that other interactions can be seen. It is through the development of the extrusion process to suit a raw material that not just allows optimisation of properties, but gives more consistent tube performance that holds greatest potential from a life modelling and prediction point of view.

The mixing of silicone has been found to be dependent on both the raw material used and the extrusion line design. Adjustment of mixing times can be used to increase life and consistency of performance from tube to tube.

The pin and die has been shown to influence the dimensional tolerance of the tubing and thus the consistency of the occlusion force that the tube is exposed to within the pump. This is of particular importance when using flow rate changes to map the normal behaviour of a tube within a system and the potential to use flow changes as part of a possible mechanism which acts as a pre-cursor to failure, as the deformation of the tube will be heavily influenced by the repeated cyclic force it is exposed to with each occlusion.

The thermal energy applied to a silicone rubber during the extrusion of a tube has been shown to influence the performance of the tube. However, more relevant is that it must be optimised by tube size and the raw material used. Therefore what is of importance to a prediction algorithm is the consistency of the thermal cycle used, along with the mix cycle, when producing a tube of certain size and material. An algorithm can then utilise empirical data gathered on the performance of tubes produced using a relatively small number of extrusion runs as a basis for the creation of standard life distribution models of 'normal' behaviour. Optimisation of the thermal cycle can be used to narrow the distribution of life, but the complexities of the cross-link process does mean that any changes to the extrusion process needs to be considered in a holistic manner, i.e. it should be considered as only a part of the manufacturing process. It is also noted that a tube's performance can only be optimised to the limit of the raw material, i.e. there is a limit to the amount of cross-linking possible in one type of raw material.

6.2.3 Pump environment

When the tube is loaded into a peristaltic pump the tube life cycle now needs to consider a number of key factors that have been shown to have a significant influence on the performance of the tube.

The temperature within which the tube is running has been shown to affect the performance of that tube. For some silicone rubbers it has been found that operating at a temperature of 4˚C is capable of increasing the average life by nearly 40% over that seen at 23˚C. At a temperature of 40˚C the average life is reduced by 10-15%. The effect of temperature is dependent on the raw material used and can be influenced by the extrusion process parameters. For example some silicone rubbers do not show the same increase in performance at the low temperature end. Here it is seen how heavily independent all aspects of the tube lifecycle become.

The system pressure to which the silicone rubber tube is exposed consistently affects the tube performance. The silicone rubbers studied were only run to 2 bar. However, the difference in life between tubes run at 2 bar and those operated at transfer pressure was significant. A drop in life of 80% was consistently observed over a range of different tube sizes and different head types. The influence of system pressure on the tube is difficult to link to the ultimate tensile strength measurement figures provided by the raw material supplier. For one silicone rubber the ultimate tensile strength was cited as 11.2N/mm^2. The hoop stress that for example a 9.6mm bore x 4.8mm wall tube is subjected to by the 2 bar system pressure is only 0.33N/mm^2. This disparity between the ultimate tensile strength value and the failure of the tubes when subjected too much lower stress values as calculated from the hoop stress highlight a much more complex stress relationship at pressure than purely a hoop stress component.

This has been borne out by the work of others (11) and points to interactions between not only stress components but also with other factors that influence life, particularly temperature, both localised and at a system level. The relationship between life and pressure for a tube size can be established clearly with a narrow pressure range using empirical data for life at transfer pressure and applying a reduction of 80%. The link between life at pressure and the size of a tube are less easy to establish. The table below shows there is rough correlation between the average life and the aspect ratio, i.e. the ratio of bore size to wall thickness, with the largest aspect ratio giving the better life figures. There is less correlation between the life performance at 2 bar and the predicted hoop stress within the tube, but there is close correlation between the predicted hoop stress and the ratio between life at pressure and life at transfer, with the tube size which shows the greatest reduction in life between that seen at transfer pressure and that seen at 2bar having the lowest theoretical hoop stress capability, see Table 29.

Average Life @ 2 bar	Tube Size Bore x wall (mm)	Aspect Ratio	Ratio of Life @ T/F pressure $v_{s.}$ 2 bar	Predicted hoop stress @ 2 bar N/mm^2
384000	9.6 x 4.8	2	5.75	0.333
373650	9.6 x 3.2	3	4.76	0.424
324000	6 x 2.4	2.5	4.64	0.378
469050	15.9 x 3.2	4.96	4.54	0.613
1296000	25.4 x 4.8	5.29	4.29	0.529

Table 29 - Comparison of life performance, hoop stress and aspect ratio for silicone tubing

It had been thought by those working within the pump industry that the lower the rotor speed and hence the lower the rate of occlusion the higher the life, with an expected linear relationship. However it has been shown that this is not the case for the silicone tubes studied. Indeed for the range of speeds analysed in detail the lower rotor speed showed the lowest fatigue life, see figure 33. This relationship remained consistent regardless of the silicone raw material used to manufacture the tubing. An algorithm therefore needs to consider rotor speed carefully when applying a predictive relationship between speed and its influence on the total life expected for a tube.

This study has utilised a wide range of pump-heads and has seen some differences between life performance for the same tube type on different heads under the same running conditions; speed, direction, temperature, pump medium etc. The reason for the difference is difficult to quantify due to the designs of the pump used in the study, where each is tailored to a range of flow rates, with little overlap between each pump. Therefore where comparisons are possible it is likely that the outer edge of the flow range is being observed, where tube performance may be thereby affected. An algorithm needs to have the pump head type as an entry. Using this method will allow the selection of correct empirical data by which expected performance can be monitored and against which it can be checked. For example a FW7 head is entered into a silicone tube based algorithm, along with a tube size of 9.6 x 4.8. Using these inputs the algorithm can recall the expected nominal average life seen in a range of conditions, the distribution of life figures expected and the flow rate per day seen with water as a pump medium. These data can be built up from historical data and then the other factors that have been shown to affect life such as system pressure, speed and temperature can then be integrated into this simple baseline model to create a more complex but more comprehensive data model.

Other factors also need to be considered, such as the medium being pumped. Classes of chemical can initially be used to produce an estimate of effect. However it is here that the learning aspect of an algorithm comes into its own. An end user can enter the life of the last tube they used in the system into an algorithm to allow the data model to 'calibrate' itself. Repeated entry will allow the analysis engine to keep learning, until it builds up knowledge of the effect of the chemical on the expected life performance. Use of single use heads or more intelligent identification systems could allow this entry by the user to be taken from a manual entry to an automated entry, removing the need for the user to enter the information.

Splitting chemicals into classes is often used in chemical compatibility tables, where an end user can assess if a tube material is viable for their process. This format also offers a basis for chemical testing of a tube material to gauge the effect on performance, so that a baseline model is more complete in terms of its knowledge base.

Other factors to consider are any sterilisation regimes used by the pump user. The effect of gamma irradiation has been shown to change the performance of silicone rubber. How the tube is affected depends on both the silicone raw material and the process used to produce the tubing. It is important that an algorithm for a silicone tube knows these aspects if it is to perform adequately.

End users and the knowledge they hold, particularly with respect to the effect of environmental factors on silicone rubber performance, could offer a good source of information. Taking a sample of end users with perhaps 'typical' applications could simplify this task in the first instance. These users of typical applications could be utilised to double-check algorithm assumptions and allow a knowledge database to improve dramatically.

6.3 System 2 – The EPDM/PP Blend tube life cycle

6.3.1 Raw Material

Data supplied by the raw material supplier allows initial material data such as shore hardness and UTS to be linked to tube performance. Characterisation tests help build the detail needed to optimise the tube performance through the manufacturing process. These tests show that when working within a specific shore hardness range, levels of filler and oil, along with the level of crosslinking within the EPDM phase are important aspects in understanding the performance of the material when used as a peristaltic pump tube.

6.3.2 Manufacturing Process

The extrusion process has the ability to alter the performance of the EPDM/PP material, both from a dimensional and a material properties point of view. Dimensional changes will affect the level of occlusion within the pump-head and therefore the level of stress incurred with each cycle and the fatigue of the tube. However dimensional changes also affect the ability of the tube to perform at pressure. If the level of occlusion is too low, the tube will not be able to contain the pillow volume under pressure with the result of a backward flow of the fluid, back-streaming, which affects the forward flow rate and which if the occlusion is too low can cause the loss of the ability of the pump to flow under high system pressures.

The extrusion process has been shown to affect the compression capability of the material. Over a range of screw speeds a high screw speed has been shown to change the material properties to give a consistently longer life tube material. A number of possible reasons for this change are cited within chapter 5, but from a predictive point of view it is the consistency of tube performance that the process optimisation brings which is the most valuable. This allows an algorithm to use a normal distribution pattern with a smaller standard deviation as a baseline model from which to apply the effect of other factors. The ability to link the raw material data more closely to the performance as an extruded tube is aided by using extruded test pieces which have been produced using the same extrusion conditions, screw speed in particular.

6.3.3 Pump Environment

With the raw material characterised and the extrusion process used to optimise the tube, as much as the material and process will allow, then the life cycle analysis turns to those factors which have a significant influence on life.

The relationship between life and pressure has been shown to be almost linear and can therefore be modelled relatively simply within an algorithm. The pressure effect is heavily influenced by the tube geometry, too low an occlusion level and back-streaming occurs as discussed earlier. It is also influenced by the shore hardness. If the shore hardness is too low for the pressure, the higher levels of EPDM phase which occur as the shore hardness is lowered mean that under high system pressure this rubber phase deforms resulting in a reduction or loss of flow, or in extreme cases permanent deformation resulting in material thinning and then ballooning leading to tube failure.

EPDM/PP blends show a reduction in life at both high and low temperatures when compared to that shown at a temperature of 23°C. This reduction is greater when combined with a high system pressure. It has been found that localised temperature in the worked area of the tube is much higher than the temperature that the tube is being subjected to. This temperature related performance is linked not only to the raw material and extrusion process used but also to any mechanical wear on the tube. The fatigue mechanism for wear on the tubes studied show that it is the polypropylene particles which make up the debris from mechanical wear induced by the peristaltic action. This results in a much greater reliance on the remaining EPDM and filler structure in resisting fatigue. It has been shown that the EPDM and filler changes from material to material even at the same shore hardness and that the extrusion process can influence the filler dispersion and EPDM.

It is perhaps rotor speed that has been shown to be the most material independent factor affecting the life of tube. It has been seen for silicone materials that low rotor speeds do not mean longer life; this is also true of the EPDM/PP blend materials. At high system pressures, such as 7 bar, life in number of occlusion cycles at 125rpm is 500% more than at 5rpm and 50% more than at 50rpm. At low speeds much greater consideration needs to be given to stress-strain curve of the material, as the roller's slow movement over the material subjects it to a greater amount of time under stress than when the rotor is moving at high speed and thus moves over the tube more quickly. The raw material and the process optimisation will affect the ability of the material to withstand this cyclic stress.

Other environmental factors to link into the life cycle to include in an algorithm have also been briefly considered. Sterilisation by gamma irradiation has been found to have a detrimental effect on EPDM/PP particularly when combined with other factors with strong influences on life, such as system pressure and temperature. Degradation of the PP phase through chain scission and changes to the EPDM crosslinking all influence the ability of the material to withstand additional fatigue cycles. Further work is needed to characterise the relationships between sterilisation techniques based on heat (autoclave) or irradiation and their effect on tube performance. Both techniques are considered detrimental to performance and further work needs to determine how detrimental within the context of interaction with other factors and how to include this within a predictive algorithm. Although as with a silicone based algorithm, entry by a user of the life of previous tubes within a set environment could allow an algorithm to calibrate its empirical data quickly to take sterilisation effects into account.

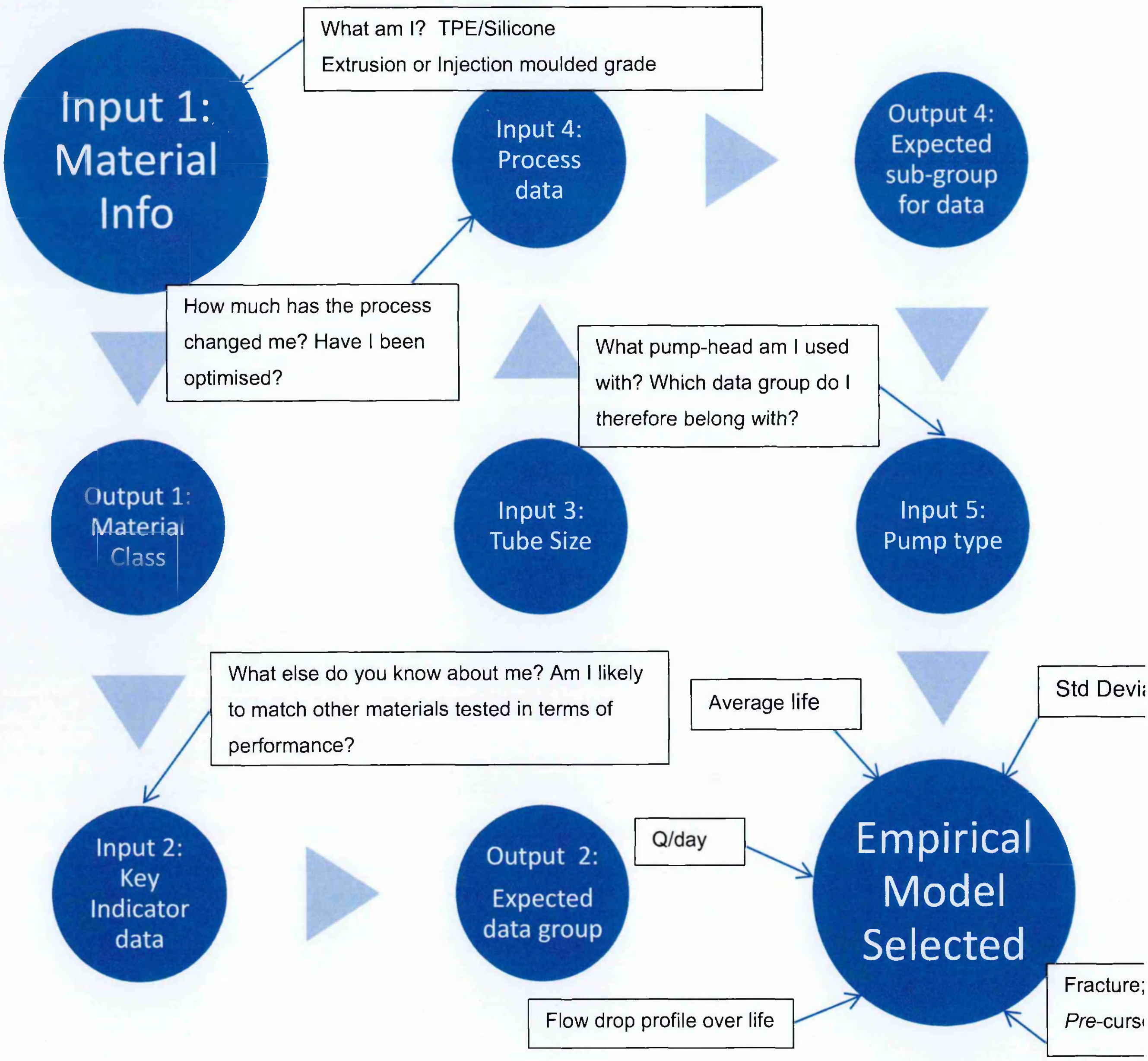
This same approach could also be used with chemicals being pumped, as put forward for silicone tubing. Chemical classifications can act as a baseline for an algorithm; however 'self-learning' is the key for improved calibration of the algorithm to the real world. Future research however should focus on commonly used chemicals used by EPDM/PP blend tube users and the specific breakdown mechanisms that occur with them. Sodium Hypochlorite, used throughout the water industry, is one such chemical. This chemical is likely to enhance oxidation of the EDPM phase, resulting in lower fatigue life. It has been noted in section 3.4.3 that the EDPM phase is a key part of the fatigue mechanism that occurs as a result of repeated occlusions. Any chemical reaction that affects the EPDM phase can therefore influence and accelerate other failure factors. It has been discussed that tube performance can be substantially affected if multiple factors which influence life are brought together, such as temperature, pressure and chemical attack. These combinational effects could be studied perhaps more fully with the knowledge and help of the end users. Typical users of sodium hypochlorite within the water industry are likely to be using similar system pressures (dosing into mains water pressure) and temperatures (in rooms where temperatures are uncontrolled and thus susceptible to global temperature patterns). Using performance data from them will help build a realistic knowledge base for an algorithm much more quickly.

7 CREATING THE ALGORITHM

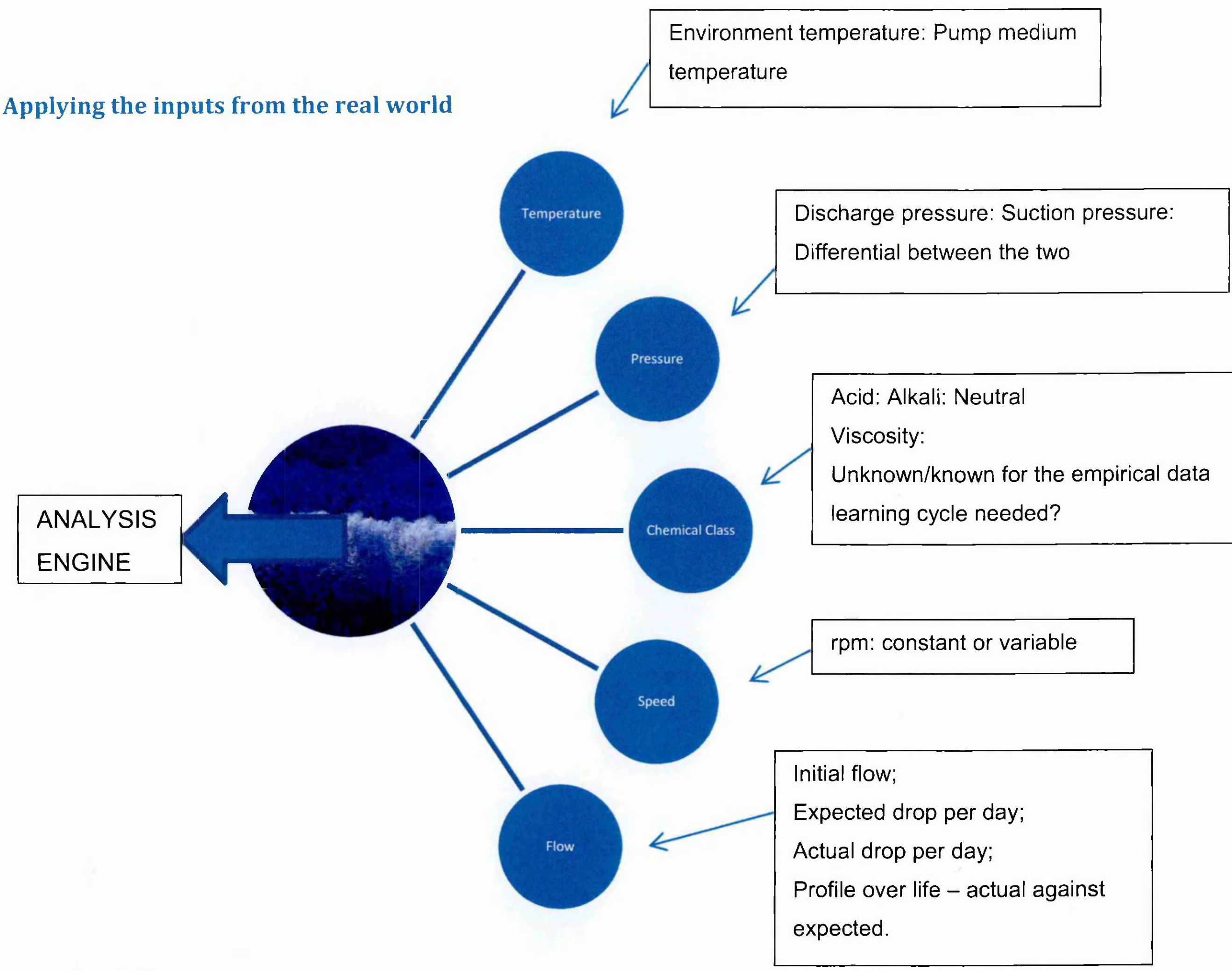
7.1 Overview

The creation of an algorithm is split into steps. First using a number of inputs the correct empirical data is chosen, where the empirical data is drawn from historical data and performance experiments. The algorithm now needs the inputs for the analysis engine: these tell it how the empirical data will need to be modified in order to produce an understanding of 'normal' performance in terms of average life and flow drop off and what may be seen as 'abnormal' or a pre-cursor to final fracture. Finally a learning cycle is applied where the algorithm can 'learn' using the life and flow figures from tubes used in the same environment to build the data on which average, standard deviation and any other distribution calculations are applied.

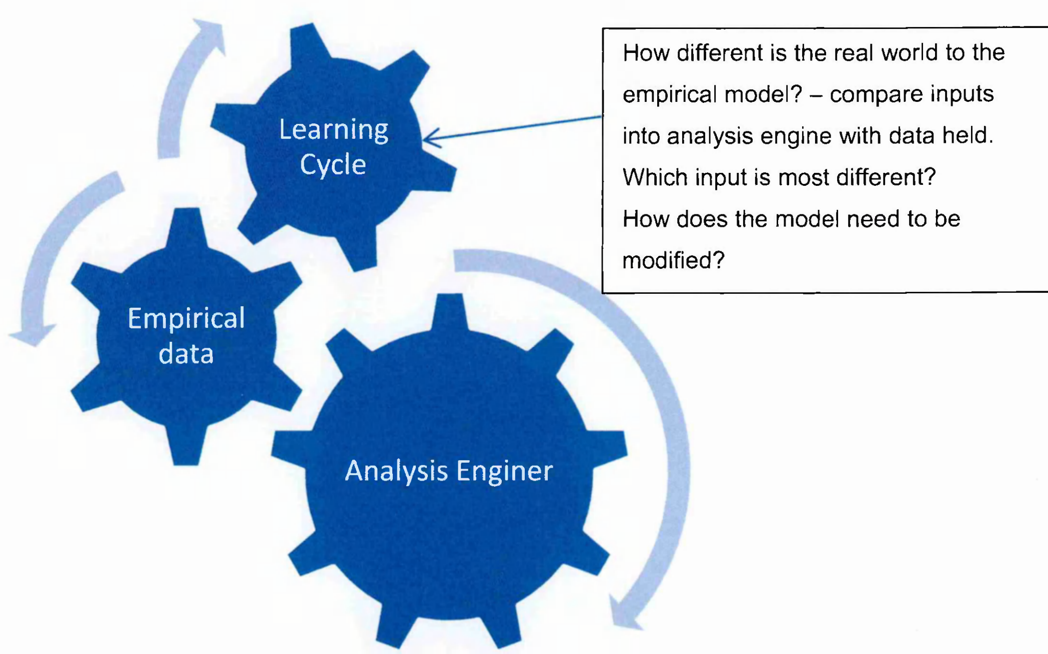
7.1.1 STEP 1: Choosing the empirical model to apply



7.1.2 Applying the inputs from the real world



7.1.3 Learning Cycle



7.2 Silicone rubber algorithm design

The process for the design for a single silicone material is drafted below.

7.2.1 Empirical model

7.2.1.1 *Input 1 – Material Information*

MATERIAL ATTRIBUTE	VALUE
Material Type	HTV Silicone
Grade	Extrusion grade
Hardness	60 Shore A
Specific Gravity	1.15
Tear B	4.2 Nmm^2
Ultimate Tensile Strength	11 Nmm^2
Elongation at Break	800%

OUTPUT 1: Material Class = S1

DESCRIPTION: HTV Silicone extrusion grade, hardness range 50 to 60 Shore A

7.2.1.2 *Input 2 – Key Indicator Data*

KEY INDICATOR ID	VALUE
Indicator 1	1.812 mg/cm^2
Indicator 2	1.1081 mg/cm^2
Indicator 3	1100 µg
Indicator 3 after irradiation	No change or decrease
Indicator 3 after thermal ageing	Decrease

OUTPUT 2: Expected Data Group = S1:1

DESCRIPTION: Silicone Group 1 – Sub Group 1

7.2.1.3 *Input 3 – Tube Size*

9.6mm bore x 2.4mm wall

7.2.1.4 *Input 4 – Process Data*

PROCESS SECTION	INFORMATION
Process Identification Number	Process 2
Screw Design	Standard 2
Hot Box Type	Standard 2
Line Oven Length	8m
CTM Mix cycle	Mix cycle optimised for this size
Thermal Cycle	Partially optimised – standard run sheet for process
Post Bake	Post bake applied

OUTPUT 3:

Material Class: Silicone S1

Data Group 1

Sub Group 9624P2 – Part Optimised

7.2.1.5 *Input 5 – Pump Type*

BW5 Pump type

7.2.1.6 *EMPIRICAL MODEL*

ATTRIBUTE	VALUE
Material Class	Silicone 1
Data Group	Sub set S1
Sub Group	Sub Group 9624P2

Pump Type	BW5
Average life expected	8943000 occlusions (based on water, 23˚C, 100rpm)
Standard Deviation expected	5133862 occlusions (based on water, 23˚C, 100rpm)
Average Flow at start of life	1387ml/min
Standard Deviation in flow at start of life	93 ml/min
Flow drop profile	3.06% over full life
Flow drop per million occlusions	0.342%

7.2.2 Entering pump inputs to the analysis engine

7.2.2.1 Pump inputs

INPUTS FROM THE REAL WORLD	VALUE
ENVIRONMENT TEMPERATURE	4˚C
SYSTEM PRESSURE	Transfer pressure
CHEMICAL	Neutral
SPEED	300rpm

7.2.2.2 Modified attribute table with new expected values using the Analysis engine

ATTRIBUTE	NEW VALUE
Material Class	Silicone 1
Data Group	Sub set S1
Sub Group	Sub Group 9624P2
Pump Type	BW5
Average life expected	11115000 occlusions
Standard Deviation expected	561087 occlusions
Average Flow at start of life	4409 ml/min average
Standard Deviation in flow at start of life	231 ml/min
Flow drop profile	8.75% over full life
Flow drop per million occlusions	0.0787%

7.2.3 Putting the third element into the model, the learning element

Questions to assess the relevance of the learning element, either by continuous assessment of sensors, i.e. pressure and flow sensors or through more detailed input by the user:

- Is the chemical being pumped really neutral?
- Is the interaction between the pump medium and the material understood?
- How many data points are needed to improve the confidence level significantly?
- Does the pressure remain at transfer throughout the life of the tube?
- Does the speed remain constant over the life of the tube?
- How does Q vary over the life of the tube, are there any discernible patterns pre-failure?
- Is the fracture position within the normal range?
- Is early onset material damage within the normal range?

7.3 EPDM/PP algorithm design

The process for the design for a single EPDM/PP material is drafted below.

7.3.1 Empirical model

7.3.1.1 Input 1 – Material Information

MATERIAL ATTRIBUTE	VALUE
Material Type	EPDM/PP
Grade	Extrusion grade
Hardness	87 Shore A
Specific Gravity	0.95
Tear B	45 $kNmm^2$
Ultimate Tensile Strength	15.6 MPa
Elongation at Break	560%
Modulus @ 100% elongation	6.80 MPa

OUTPUT 1: Material Class = TPV1

DESCRIPTION: EPDM/PP extrusion grade, hardness range 84 to 95 Shore A

7.3.1.2 Input 2 – Key Indicator Data

KEY INDICATOR ID	VALUE
Indicator 1	0.4 Silica / 0.21 Crystalline PP
Indicator 2	24.7 non-rubber component
Indicator 3	48.8
Indicator 4	NMR 0.88
Indicator 5	TEM snap shots show morphology similar to data held

OUTPUT 2: Expected Data Group = TPV1:1

DESCRIPTION: Process applicable changes to be checked

7.3.1.3 Input 3 – Tube Size

6.4mm bore x 3.2mm wall

7.3.1.4 Input 4 – Process Data

PROCESS SECTION	INFORMATION
Process Identification Number	Process 1
Screw Design	Standard 1
Extruder Speed	64rpm
Extruder Temperature Profile	175/180/185/190˚C
Extruded dog-bone tensile results	Modulus @ 100% 5.7MPa
Tubing compression results	32kg @ 60 minutes
Tubing swell results	1.24

OUTPUT 3:

Material Class: Silicone S1

Data Group EP1

Sub Group 6432P1 – Optimised

7.3.1.5 Input 5 – Pump Type

CWN Pump type

7.3.1.6 EMPIRICAL MODEL

ATTRIBUTE	VALUE
Material Class	TPV1
Data Group	EP1
Sub Group	Sub Group 6432P1
Pump Type	CWN
Average life expected	51million occlusions (based on water, 23˚C, 100rpm)

Standard Deviation expected	7.6million occlusions (based on water, 23˚C, 100rpm)
Average Flow at start of life	398 ml/min
Standard Deviation in flow at start of life	37 ml/min
Flow drop profile	21% over full life
Flow drop per million occlusions	0.2055%

7.3.2 Entering pump inputs to the analysis engine

7.3.2.1 Pump inputs

INPUTS FROM THE REAL WORLD	VALUE
ENVIRONMENT TEMPERATURE	40˚C
SYSTEM PRESSURE	7 bar
CHEMICAL	Neutral
SPEED	125rpm

7.3.2.2 Modified attribute table with new expected values using the Analysis engine

ATTRIBUTE	NEW VALUE
Material Class	TPV1
Data Group	EP1
Sub Group	Sub Group 6432P1
Pump Type	CWN
Average life expected	12.127million occlusions
Standard Deviation expected	720000 occlusions
Average Flow at start of life	505 ml/min
Standard Deviation in flow at start of life	23ml/min
Flow drop profile	19% over full life
Flow drop per million occlusions	1.566%

7.3.3 Putting the third element into the model, the learning element

Questions to assess the relevance of the learning element, either by continuous assessment of sensors, i.e. pressure and flow sensors or through more detailed input by the user:

- Is the chemical being pumped really neutral?
- Is the interaction between the pump medium and the material understood?
- How many data points are needed to improve the confidence level significantly?
- Does the pressure remain at 7 bars throughout the life of the tube?
- What is the differential between the suction and the discharge pressure readings
- Does the speed remain constant over the life of the tube?
- How does Q vary over the life of the tube, are there any discernible patterns pre-failure?
- Is the fracture position within the normal range?
- Is early onset material damage within the normal range?

8 CONCLUSIONS

8.1 Overview

Life cycle analysis has been carried out for tubes produced in two materials considered important within the peristaltic pump industry. Raw material information has been enhanced through the use of key material indicators. The tube manufacturing process has been considered and the influence it has on the performance of the tube within the pump. Finally the effect of those environmental factors that influence life has been investigated, with typical tube fractures and positions being shown.

The study allows a first draft algorithm design example for both materials to be created. From this work a number of conclusions have been reached.

8.2 General conclusions

- The performance of a tube within a peristaltic pump is as a result of complex interactions between the material, the process used to produce the tube, the pump it is in and the environment to which it is subjected. No part of the life cycle should be considered in isolation.
- The stresses to which the tube is subjected are also complex and there has been only limited success modelling these stresses. However, it has been found through previous studies that the shear component with the stress model does seem to play a key part in fracture of the tube wall.
- Tubes within a peristaltic pump show material changes over time, this is most noticeable to the end user through the change in flow rate, Q of the medium being pumped. The change in flow rate has been shown to be directly linked to tube material changes and indeed is a good indicator of a tubes health.
- It has been shown that both material types studied can be optimised in terms of their performance within a peristaltic pump, through material selection, process optimisation and tube size tolerance.
- Pump-head types have been shown to have significant differences in average life and standard deviation. The flow change profiles for the heads studied were shown to be noticeably different. It is apparent that this difference needs to be clearly incorporated into an algorithm so that changes in Q can be analysed correctly.
- Although there is a large amount of data gathered it is clear that a knowledge based algorithm for prediction requires much more data than could be gathered in this study. It is therefore understood that

the learning part of the algorithm is vital for efficient algorithm development. The learning can be through inputs from the user or data gathering by the pump itself through non-invasive measurement, data collection and analysis.

8.3 Conclusions specific to Silicone materials

- It is possible with silicone to optimise the network structure through effective cross-linking both through material choice and manufacturing process parameters.
- Material choice is vital if the material is to be optimised for a peristaltic pump; high silica content in the region of 28 to 30% would appear to be very beneficial for tube performance in the environments studied, however this loading is only thought to be appropriate if the silica particle has been optimised for good crosslinking whilst still being in the region where the material can be extruded effectively. Good tube performance was linked to high cross-linking.
- Key indicators have shown that a known set of experiments may be used to show where optimised performance is likely and to compare similar silicone and thus choose the most appropriate for the application.
- Tube size has been shown to be vital for process optimisation, both in terms of the dimensional tolerance which influences occlusion gap and also the amount of thermal energy imparted to the material.
- Injection moulded silicone tubes which use LSR perform much better in terms of life than extruded silicone rubber tubes. They have been shown to have much greater levels of cross-linking. How this level of cross-linking can be transferred to the solid HTV silicone material types used in extrusion processes needs further research. Indeed, material scientists at the manufacturers which supplied some material used in this study are already looking at this area.
- Environmental factors which affect life have been found. Pressure was found to have a huge influence on life, with life at 2 bar only 20-25% of the life at transfer pressure. The effect of temperature on silicone rubber was interesting, for some silicone materials a low temperature of 4°C would increase life by 40%; however this was not seen in all the silicone rubbers studied. The life at a high temperature of 40°C was consistently reduced by 10-15% from the life seen at 23°C. The speed that the tube is run at also affected the life seen, with an optimum number of occlusions seen at higher speeds.
- The use of pre-cursors to failure have had limited success for silicone. The silicone rubbers studied were only for 0 to 2 bar pump pressure, with the high percentage of application being around transfer (0

to 0.5 bars). Therefore the use of pressure traces to look for any discernible patterns in behaviour is inappropriate. When the pump type is known it may be effective to utilise flow profiles to look for patterns of pre-failure behaviour, however it was felt that there was not enough high quality data in this area to effectively utilise this approach at this stage.

8.4 Conclusions specific to EPDM/PP materials

- It is possible with the EPDM/PP material to optimise the manufacturing process to give the most consistent tube performance by monitoring the EPDM cross-linking through a combination of swell tests and compression testing. The section of the manufacturing process found to have the greatest influence was the combination of screw speed with extruded line run rate. At higher screw rates the EPDM/PP material displayed the most consistent performance, offering the greatest opportunity for an algorithm to be reliably used.
- Key indicator tests can be used to look at comparable EPDM/PP materials and the results can be linked to key material characteristics and tube performance. The key indicator tests for EPDM/PP materials were seen to act in a different way to the silicone materials. Their use was more limited and they were used as confirmation tests for similar materials. The process influences on material properties were found to be far more influential in optimising the performance.
- Environmental factors which affect life have been found. Pressure was found to have a huge influence on life, with life at 7 bar only 20-25% of the life at transfer pressure. The effect of temperature on the EPDM/PP blends showed that both a low temperature of 4˚C and a high temperature of 40˚C would consistently reduce the life seen at a 23˚C. At 40˚C this was by over 70% and at 4˚C by 30%. The speed that the tube is run at also affected the life seen, with an optimum number of occlusions see at higher speeds.
- The use of pre-cursors to failure have had limited success. At high pressure it has been seen that the differences between the discharge and suction pressure may offer opportunities for modelling pre-cursor failure, but at transfer pressure this same approach does not offer perceivable patterns to enable modelling. As with the silicone material when the pump type is known it may be effective to utilise flow profiles to look for patterns of pre-failure behaviour, however it was felt that there was not enough quality data in this area to effectively utilise this approach at this stage.

8.5 Recommended further work

It has been found that data held in some areas is non-existent or weak and this, along with other areas, offer opportunities for further work to enhance this area of study:

- Chemical testing on tubing across a broad range of chemicals to build up better empirical data from which to draw on for the empirical model. Chemicals should be chosen which represent the key usage with industry thus enabling the model to further know about the 'real world'.
- Use acoustic emissions as a non-invasive method to monitor crack propagation. This can then be mapped to flow profiles and pressure traces to produce a performance picture, particularly at transfer pressure, against which empirical data is checked. The algorithm can then learn more quickly about how much it needs to modify the analysis engine before it is modelling normal behaviour correctly.
- Greater analysis of the pump-head design to optimise performance further, through reduction of occlusion stresses to the absolute minimum to maintain flow. Perhaps through design of pump-heads which are more specific for a narrower flow range, something some pump manufacturers are doing.
- Learning cycle enhancements to prevent the need for user input of data. For example the use of communication technology so data can be passed from pump-head or tube to the pump when a tube fails, thus enabling the pump to gather data quickly and unobtrusively on tube behaviour in an application. This would greatly enhance what is a knowledge based algorithm.
- Including the end user in the data collection for an algorithm has been discussed briefly in previous chapters. This area offers huge potential to bring real application data into the knowledge database, particularly the effects of combinational degradation. This would allow an algorithm to use live data to learn from and allowing any experimental data to be checked against.
- Proving the algorithm under both experimental conditions and in selected end user's specific applications.

9 APPENDICES

9.1 Appendix 1: Certificate of Analysis for Silicone rubber

Characteristic	Unit	Result	Lower Limit	Upper Limit
A: | FE-3: SHORE A Method QM02 MDA01316 / 1 SHORE		63.0	58.0	68.0
A: | FE-1: TENS PSI Method QM02 MDA01314 / 1 TENSILE	PSI	1300	1100	
A: | FE-1: ELONG % Method QM02 MDA01312 / 1 ELONGATION	%	766	350	
A: | FE-19: TEAR B PPI Method QM02 MDA01315 / 1 TEAR	PPI	296	175	
A: | FE-1: MOD@250 % Method QM02 MDA01313 / 1 MODULUS		565	450	
A: | E-11: COMP SET % Method QM02 MDA01147 / 1 COMPRESSION SET	%	17.56		50.00
A: | E-2: APPEAR Method QM02 MDA01172 / 1 APPEARANCE				

Characteristic	Unit	Result	Lower Limit	Upper Limit
A: | C-1028: IP SP GRAV Method QM02 MDA00123 / 1 SPECIFIC GRAVITY		1.180000	1.166000	1.191000

9.2 Appendix 2: Certificate of Analysis for EPDM/PP blend

Elastomers	Typical Value (English)	Typical Value (SI)	Test Based On
Tensile Strain at Break - Across Flow (73°F (23°C))	560 %	560 %	ISO 37
Tear Strength - Across Flow			ASTM D624
73°F (23°C), Die C	257 lbf/in	45.0 kN/m	
212°F (100°C), Die C	143 lbf/in	25.0 kN/m	
Tear Strength - Across Flow			ISO 34-1
73°F (23°C), Method Bb, Angle (Nicked)	260 lbf/in	45 kN/m	
212°F (100°C), Method Bb, Angle (Nicked)	140 lbf/in	25 kN/m	
Compression Set			ASTM D395B
73°F (23°C), 168 hr, Type 1	33 %	33 %	
212°F (100°C), 168 hr, Type 1	48 %	48 %	
Compression Set			ISO 815
73°F (23°C), 168 hr, Type A	33 %	33 %	
212°F (100°C), 168 hr, Type A	48 %	48 %	

Physical	Typical Value (English)	Typical Value (SI)	Test Based On
Specific Gravity	0.950	0.950	ASTM D792
Density	0.950 g/cm^3	0.950 g/cm^3	ISO 1183

Hardness	Typical Value (English)	Typical Value (SI)	Test Based On
Shore Hardness			ISO 868
Shore A, 73°F (23°C), 0.0787 in (2.00 mm)	94	94	

Elastomers	Typical Value (English)	Typical Value (SI)	Test Based On
Tensile Set (73°F (23°C))	28 %	28 %	ASTM D412
Tensile Set (73°F (23°C))	28 %	28 %	ISO 2285
Tensile Stress at 100% - Across Flow (73°F (23°C))	986 psi	6.80 MPa	ASTM D412
Tensile Stress at 100% - Across Flow (73°F (23°C))	986 psi	6.80 MPa	ISO 37
Tensile Strength at Break - Across Flow (73°F (23°C))	2260 psi	15.6 MPa	ASTM D412
Tensile Stress at Break - Across Flow (73°F (23°C))	2260 psi	15.6 MPa	ISO 37
Elongation at Break - Across Flow (73°F (23°C))	560 %	560 %	ASTM D412

9.3 Appendix 3: Example of a production run sheet for extrusion of silicone rubber

RUN SHEET				REV :2
DATE		QA143	Issue:1	
EXTRUDER mm		W/O		
TOOLING	Pin number	Die number		
SCREEN PACKS				
Tube size:	mm bore x		mm wall	
Nominal OD mm	Maximum OD mm		Minimum OD mm	

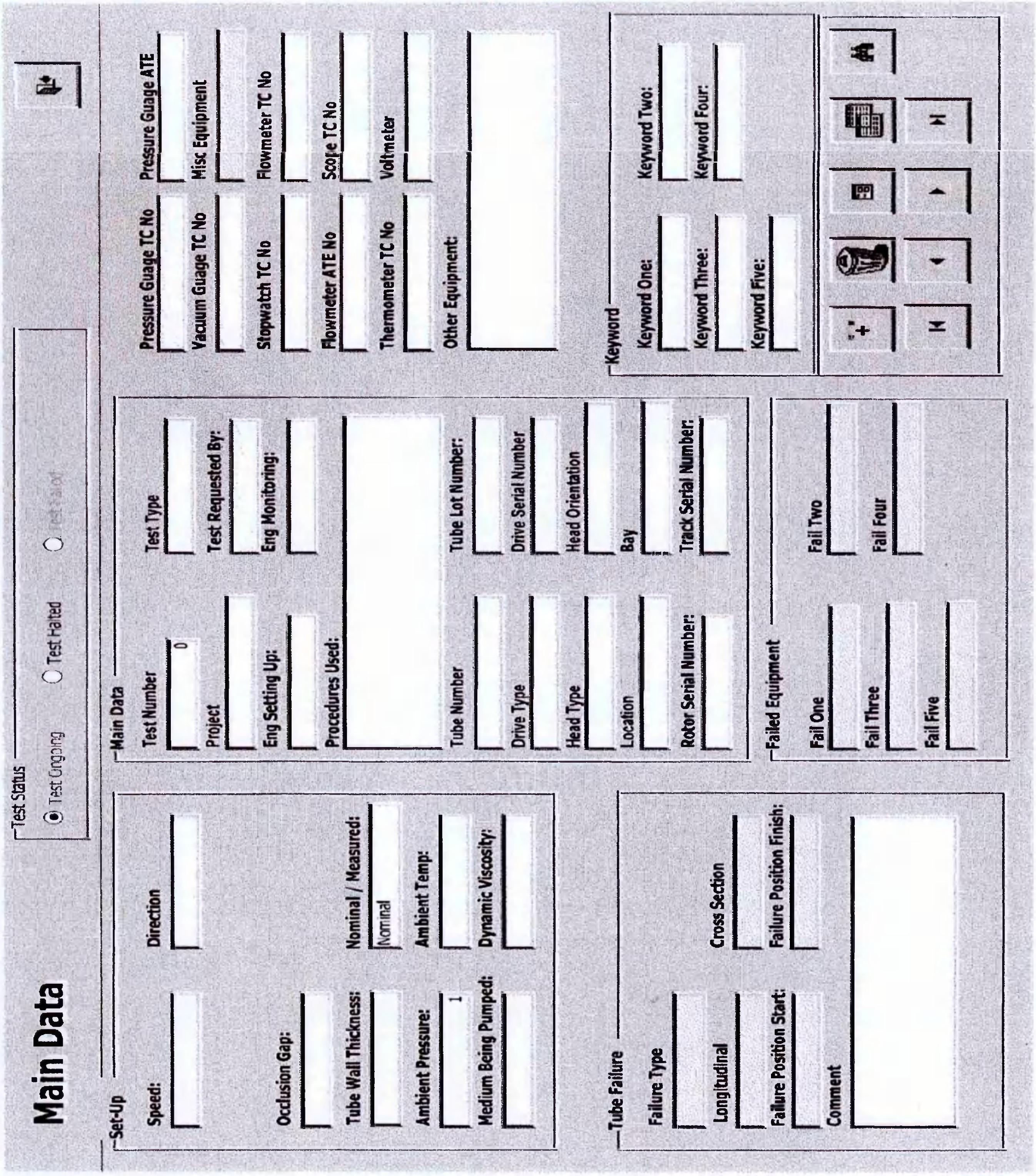
CTE 'K VALUE'		Works order
EXTRUDER HEAD TEMP		
EXTRUDER CYLINDER 1 TEMP		Partcode
EXTRUDER CYLINDER 2 TEMP		
EXTRUDER FEEDING TEMP		Lot number
SCREW TEMP		
CTE BODY TEMPERATURE		
EXTRUDER PRESSURE		
RUN RESULTS		
TIME		Hot box number TC number
HOTBOX TEMPERATURE		
TUBE SURFACE TEMPERATURE °C		
DIE PRESSURE		
OFFSET		
LINE OVEN SPEED		
LINE OVEN ZONE 2 TEMP		
LINE OVEN 2 UPPER FAN RPM		
LINE OVEN 2 LOWER FAN RPM		
LINE OVEN ZONE 1 TEMP		
LINE OVEN 1 UPPER FAN RPM		
LINE OVEN 1 LOWER FAN RPM		
105mm Line Only		
LINE OVEN ZONE 3 TEMP		
LINE OVEN 3 UPPER FAN RPM		
LINE OVEN 3 LOWER FAN RPM		
PACKAGING FILM		
FILM LOT NUMBER		
POST CURE OVEN		

	Signature	COMMENTS
Set by:		
Set Check by:		
Laser code set		
Laser code check		
Proving run		
Lot Number		
Graph saved		
Coiling lot no check by:		
Streaming level and appearance OK		
Tooling cleaned, checked and put back in location by:		

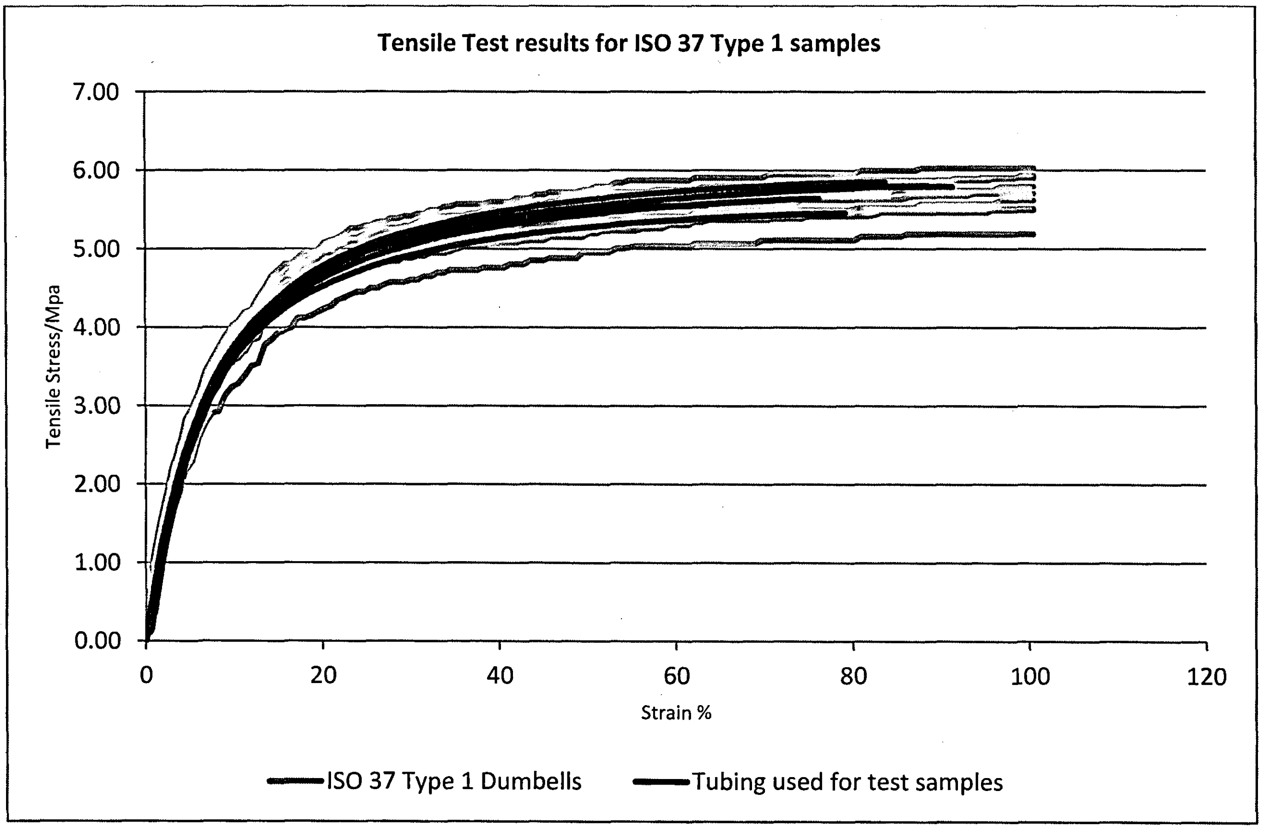
9.4 Appendix 4: Example of production run sheet for extrusion of EPDM/PP blends

32mm LINE RUN SHEET		Date		V3 24/01/2005
Tubing lot number				
Tube bore:	mm	Tube wall:	mm	
Bore min	mm	OD min	mm	
Bore max	mm	OD nom.	mm	
Wall nom	mm	OD max	mm	
Material		Hardness		
Tooling				
Pin number	mm	Pin size	mm	
Die number	mm	Die Size	mm	
Screen packs				
Vacuum bath				
Distance from die to weir inlet				mm
Wier plate number		Wier plate diameter		mm
Entry plate number		Entry plate diameter		mm
Sizing plate number		Sizing plate diameter		mm
Second water bath				
Discharge seal number		Discharge seal size		mm
Air knife number		Air knife size		mm
Cutter				
Cutter bush number		Cutter bush size		mm
Cutter blade type		Cutter blade mounting (description)		
Run conditions				
Barrel zone 1 temperature			^{o}C	
Barrel zone 2 temperature			^{o}C	
Barrel zone 3 temperature			^{o}C	
Barrel zone 4 temperature			^{o}C	
Clamp temperature			^{o}C	
Head temperature			^{o}C	
Die temperature			^{o}C	
Melt pressure			bar	
Melt temperature			^{o}C	
Extruder speed			rev/min	
Extruder current			amps	
Haul off speed			m/min	
Vacuum			bar	
Lasermike control gain				
Lasermike comments				
Water temperature vacuum bath			^{o}C	
Water temperature second bath			^{o}C	

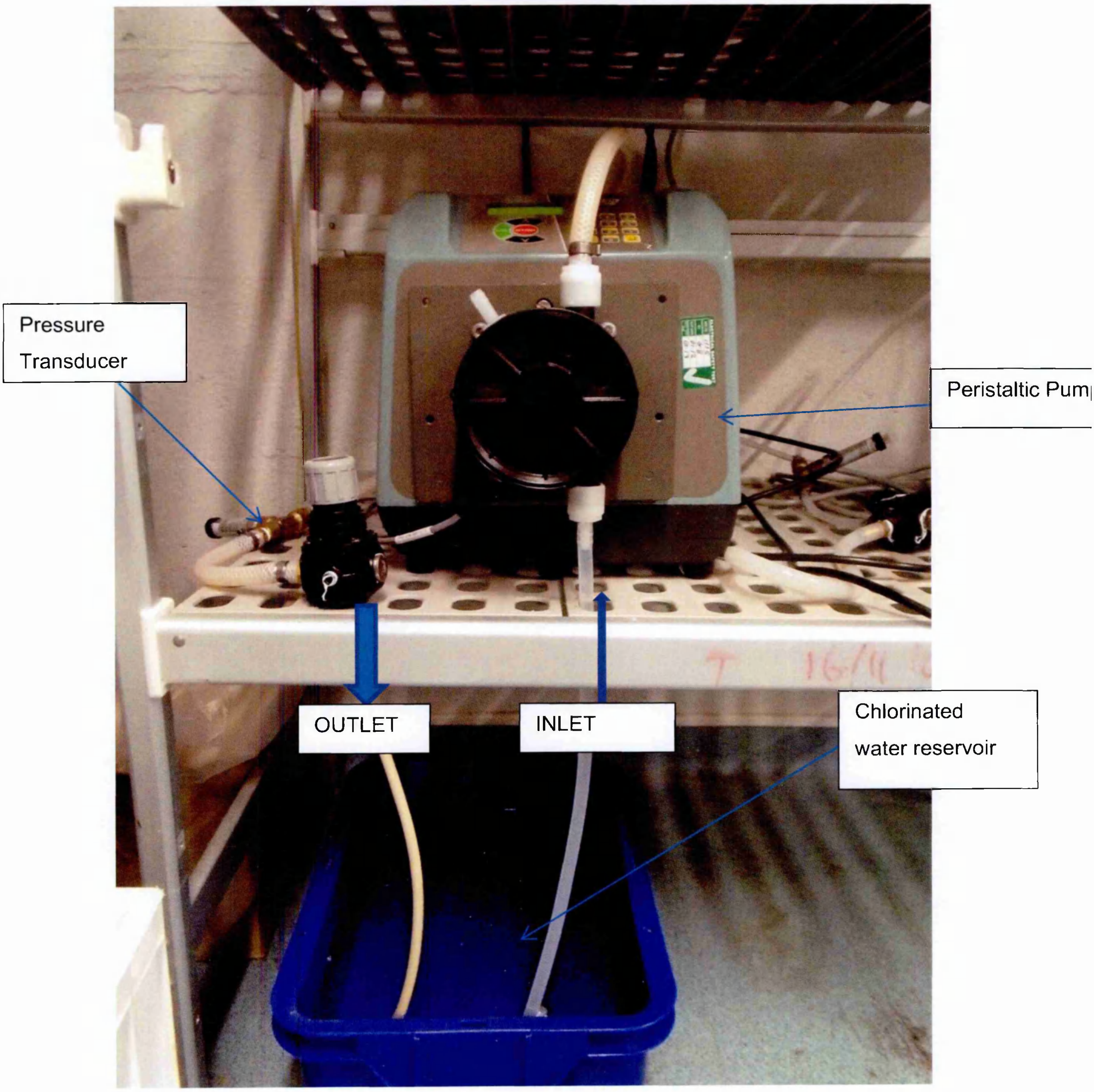
9.5 Appendix 5: Data entry sheet for database logging of experiments



9.6 Appendix 6: EPDM/PP Dog bone vs. tubing test results



9.7 Appendix 7: Standard test station layout



9.8 Appendix 8: Observations of failure for silicone rubber tube

COLOUR CODE KEY:			
	4.8mm bore x 1.6mm wall		0.5mm bore x 1.6mm wall
	8.0mm bore x 1.6mm wall		6.4mm bore x 2.4mm wall
	1.6mm bore x 2.4mm wall		
	9.6mm bore x 2.4mm wall		

Database No	Fracture Position	No of hours run	Number of occlusions	Head Type	Environmental Conditions: Temp	Speed	Pressure	Dir	Chevrons Top Surface (Non-break thro)	Top Surface abrasion - chevron break thro'	Scalloping	Bottom Surface Shiny	Voids within tube wall	Surface crazing/micro-cracking at cheek edge	Micro-Cracking formed fracure	Internal Orange peel damage	Fracture Area	Tube Bore	Tube Wall	Tube AR	Avg Swell Results
2344	Outer edge, discharge end	58	2088000	FW5	23°C	300rpm	T/F	CW	Y	N	Bottom	Less shiny than top	Both sides	Top and Bottom	Y	Y	Micro-Void break thro from outer to inner	4.8	1.6	3	1.63
2319	Outer edge, discharge end	70	2520000	FW5	23°C	300rpm	T/F	CW	Y (Less prevalent han 2344)	N	Bottom	Less shiny than top	Both sides - less prevalent than 2344	Top and Bottom	Y	Y	Micro-Void break thro from outer to inner	4.8	1.6	3	1.58
2293	Inner edge, mid sweep	96	3456000	FW5	23°C	300rpm	T/F	CW	Y	N	Bottom	Less shiny than top	Both sides	Top and Bottom	Y	Y	Micro-Void break thro from outer to inner	4.8	1.6	3	1.55
2333	Inner edge, discharge end	53	1908000	FW5	23°C	300rpm	T/F	CW	Y	N	Bottom	Less shiny than top	Both sides	Top and Bottom	Y	Y	Micro-Void break thro from outer to inner	4.8	1.6	3	1.56
2337	Outer edge, discharge end	85	3060000	FW5	23°C	300rpm	T/F	CW	Y	N	Bottom	Less shiny than top	Both sides, but particularly inner cheek	Top and Bottom	Y	Y	Micro-Void break thro from outer to inner	4.8	1.6	3	1.55
2349	Outer edge, discharge end	125	3300000	FW5	40°C	220rpm	T/F	CW	Y	N	On Outer edge on cheek	Bottom Surface much shinier than top	Large voids thro length of tube along cheek	N	N	Y	Void break thro from inner to outer	8	1.6	5	1.51
2334	Outer edge, discharge end	121	3194400	FW5	40°C	220rpm	T/F	CW	Y - raised mat'l @ discharge end	N	Outer edge	Bottom Surface much shinier than top	Large voids thro length of tube along cheek	N	N	Y	Void break thro from inner to outer	8	1.6	5	1.66
2317	Inner edge, discharge end	145	3828000	FW5	40°C	220rpm	T/F	CW	Y	N	Inside edge - Mid sweep	Bottom Surface much shinier than top	Large voids thro length of tube along cheek	Y	N	Y	Void break thro from inner to outer	8	1.6	5	1.53
2343	Outer edge, discharge end	135	3564000	FW5	40°C	220rpm	T/F	CW	Y - not heavily pronounced	N	N	Bottom Surface much shinier than top	Quite small no fo mid and large voids thro length of tube along cheek	Y - bottom cheek edge	N	Y	Fracture site vertical void growth from inside to outside	8	1.6	5	1.47
2302	Outer edge, discharge end	94	2481600	FW5	40°C	220rpm	T/F	CW	N	N	N	Bottom Surface much shinier than top	Different void pattern to others in exp set.	Y	Y	N	Fracture site micro-void growth from outside to inside.	8	1.6	5	1.58
2361	#	212	5596800	FW5	23°C	220rpm	T/F	CW	N	Y	N	Bottom Surface much shinier than top	Voids thro length of tube.	Y	Y	Y		1.6	2.4	0.66	1.58
2356	#	242	6388800	FW5	23°C	220rpm	T/F	CW	N	Top surface heavily pitted, with micro lateral/vertical striations/cracks	N	Bottom Surface much shinier than top	Numerous voids throughout the tube, micro-voids & crazing evident on cheeks	Y	Y	Y		1.6	2.4	0.66	1.56
2359	#	365	9636000	FW5	23°C	220rpm	T/F	CW	N	N	Y - Top heavily scalloped	Bottom Surface much shinier than top	Numerous larger voids throughout the tube	N		Y	Growth of voids from inside to outside most prevalent	1.6	2.4	0.66	1.55
2329	Inner edge, discharge end	270	7128000	FW5	23°C	220rpm	T/F	CW	N	N	Y - Top heavily scalloped	Bottom Surface much shinier than top	Distinct damage on inner bore, voids from inner bore growing thro-out length of tube, voids also separate to bore.	Y	N	Y	Fracture growth from inner bore to outer.	1.6	2.4	0.66	1.63
2292	Outer edge, discharge end	244	6441600	FW5	23°C	220rpm	T/F	CW	N	N	Y - Bottom heavily scalloped	N	Distinct damage on inner bore, voids from inner bore growing out thro-out length of tube	Y - bottom cheek edge	Y	N		1.6	2.4	0.66	1.55
2373	Multiple - see damage	314	3768000	FW5	23°C	100rpm	T/F	CW	Y	N	N	Bottom Surface much shinier than top	Considerable damage along both sides on the cheeks. Including vertical straitions	Y	Y	Y	Fracture site is growth from microvoids along line of cheek edge into the tube inner bore	9.6	2.4	4	1.68
2369	#	292	3504000	FW5	23°C	100rpm	T/F	CW	N	N	N	Bottom Surface much shinier than top	Y	Y	Y	Y	Fracture on cheeks along length of tube on outside. Vertical striations also present as fracture sites. Vertical striations also present as fracture sites	9.6	2.4	4	1.5
2331	#	227	2724000	FW5	23°C	100rpm	T/F	CW	N	Y	N	Bottom Surface much shinier than top	Y	Y	Y	Y	Fracture at discharge end on inner edge., side split micro-void growth from outside to inside.	9.6	2.4	4	1.54
2555	Discharge end, inner and outer edge.	206	2472000	FW5	23°C	100rpm	T/F	CW	N	Y	N	Bottom Surface much shinier than top	Y	Y	Y	Y	fracture growth from micro-voids from outside to inside	9.6	2.4	4	1.52
2367	Discharge end, outer edge.	167	2004000	FW5	23°C	100rpm	T/F	CW	N	Y	N	Bottom Surface much shinier than top	Y	N	N	Y	Fracture site is at discharge end, outer edge. Horizontal split along tube cheek on bottom, growth from outside to inner. Vertical straitions near fracture site.	9.6	2.4	4	1.57
2323	#	433	5196000	FW5	4°C	100rpm	T/F	CW	N	Y	Y - Bottom heavily scalloped	N	Top surface heavily pitted with vertical straitions along half of tube from mid-sweep to discharge	N	N	N	Fracture seems to be outside to inside along one of these vertical straitions	0.5	1.6	0.3125	1.53
2360	Fracture discharge end. Outside cheek, bottom surface	501	6012000	FW5	4°C	100rpm	T/F	CW	N	Y	Same scalloping along bottom surface as 2323. half-way along tube from mid-sweep to discharge	N	Heavy vertical straitions on top surface,	N	N	N		0.5	1.6	0.3125	1.44
2378	Discharge end, outer edge	381	4572000	FW5	4°C	100rpm	T/F	CW	N	Y	Same scalloping along bottom surface as 2323. half-way along tube from mid-sweep to discharge	N	Heavy vertical straitions on top surface,	N	N	N	vertical striation from outside to inside @ discharge end	0.5	1.6	0.3125	1.71

2377	#	390	4680000	FW5	4°C	100rpm	T/F	CW	Y	N	N	Bottom Surface much shinier than top	Y	Y	N	N	Fracture really difficult to spot.	0.5	1.6	0.3125	1.54
2399		327	3924000	FW5	4°C	100rpm	T/F	CW	N	top surface shows vertical striation damage mid sweep towards discharge end.	N	Bottom Surface much shinier than top	Y	N	N	N	Fracture site is vertical striation outside to insde growth	0.5	1.6	0.3125	1.56
[illegible]	Discharge end, inner and outer edge.	267	7048800	FW5	4°C	220rpm	T/F	CW	Y	N	N	N	These micro-voids have joined to form a longitudinal, horizintal crack. Voids and streaming apparent in tube outside stress area.	Y	These micro-voids have joined to form a longitudinal, horizintal crack.	Y		6.4	2.4	2.66	1.65
[illegible]	Discharge end, inner edge.	620	16368000	FW5	4°C	220rpm	T/F	CW	N	N	Sunstantial tramlining on top surface along both cheek edges, inner and outer.	Silicone residue on the bottom surface of the tube.	Y	Y	N	Substantial inner bore damage, particularly on top inner surface @discharge end.	Fractures are vertical growth traversing cheek	6.4	2.4	2.66	1.46
[illegible]	Mid sweep and discharge end, inner and outer edges.	294	7761600	FW5	4°C	220rpm	T/F	CW	N	N	N	Bottom surface heavily deposted with silicone, when removed by thumb nail, surface slightly pitted.	Voids apparent in tube in un-stressed area 0.2mm dis approx	Edge of cheeks sustained most damage, roughening of surface, voids, cracks and fracture along full length of tube	N	Y	Crack growth from lower part of cheek into tube wall. Multiple fractures along outer and inner cheek	6.4	2.4	2.66	1.49
[illegible]	Discharge end, inner edge.	228	6019200	FW5	4°C	220rpm	T/F	CW	N	N	N	N	Voids apparent thro' tube, not only in deformation area	Edge of cheeks sustained most damage, roughening of surface, voids, cracks and fracture along full length of tube	N	Inner bore damage apparent, orange peel @discharge end	Growth from outside to insude horizontally on top cheek edge.	6.4	2.4	2.66	1.45
[illegible]	Discharge end, inner and outer edge.	192	5068800	FW5	4°C	220rpm	T/F	CW	N	N	N	Some silicone residue depositted on bottom surface.	Voids apparent in tube in un-stressed area.	Along cheeks, horizontal crack formation occurs mainly on the outer edge.	Y	N	growth of cracks mainly from top cheek edge down, but also showing growth from bottom cheek edge up.	6.4	2.4	2.66	1.46
2301	Discharge end, inner edge.	169	6084000	FW5	40°C	300rpm	T/F	CW	Y	N	Severe scalloping on bottom surface and on inner edge.	N	.Voids showing in un-used area of tube.Mutiple voids and cracks showing thro' length of tube.	N	N	Y	Fracture was growth from inner bore to outer wall	1.6	2.4	0.66	1.51
2321	Mid sweep, inner edge.	142	5112000	FW5	40°C	300rpm	T/F	CW	Distinct chevrons on top surface along length of tube in pump head.	N	Scalloping on bottom surface, tramlining on top surface, this scallop position is the same on all the 1.6 x 2.4 tubes run in this exp set.	Some silicone residue on bottom surface.	Multiple cracks and voids along length of tube. Tiny voids apparent in un-used sections of tube.	N	N	Y		1.6	2.4	0.66	1.52
2335	Discharge end, inner edge.	155	5580000	FW5	40°C	300rpm	T/F	CW	Distinct chevrons on top surface along length of tube in pump head.	N	Scalloping on bottom surface, tramlining on top surface, this scallop position is the same on all the 1.6 x 2.4 tubes run in this exp set.	Some silicone residue on bottom surface.	Multiple cracks and voids along length of tube. Tiny voids apparent in un-used sections of tube.	N	N	Y		1.6	2.4	0.66	1.51
2342	Discharge end, inner edge.	127	4572000	FW5	40°C	300rpm	T/F	CW	Distinct chevrons on top surface along length of tube in pump head.	N	Scalloping on bottom surface, tramlining on top surface, this scallop position is the same on all the 1.6 x 2.4 tubes run in this exp set.	Some silicone residue on bottom surface.	Multiple cracks and voids along length of tube. Tiny voids apparent in un-used sections of tube.	N	N	Y		1.6	2.4	0.66	1.49
2345	Discharge end, inner edge.	164	5904000	FW5	40°C	300rpm	T/F	CW	Distinct chevrons on top surface along length of tube in pump head.	N	Scalloping on bottom surface, tramlining on top surface, this scallop position is the same on all the 1.6 x 2.4 tubes run in this exp set.	Some silicone residue on bottom surface.	Multiple cracks and voids along length of tube. Tiny voids apparent in un-used sections of tube.	N	N	Tiny voids apparent in un-used sections of tube. Inner bore damage apparent thro' length of tube. Some silicone residue on bottom surface.		1.6	2.4	0.66	1.53
[illegible]	Discharge end, inner and outer edge.	151	1812000	FW5	40°C	100rpm	T/F	CW	N	N	N	Bottom surface very smooth, no residue.	Multiple cracks and voids along length of tube. Tiny voids apparent in un-used sections of tube.	Y	Y	Y	Fracture horizontal crack growth on top cheek edge, growth outer bore to inner bore, from collection of smaller voids	6.4	2.4	2.66	1.46
[illegible]	Discharge end, outer edge. (Sites also mid-sweep & suction end)	200	2400000	FW5	40°C	100rpm	T/F	CW	N	N	N	Bottom surface very smooth, no residue.	Multiple cracks and voids along length of tube. Tiny voids apparent in un-used sections of tube.	Y	Y	N	Fracture horizontal crack growth on top cheek edge, growth outer bore to inner bore, from collection of smaller voids	6.4	2.4	2.66	1.36
[illegible]	Discharge end, inner and outer edge.	232	2784000	FW5	40°C	100rpm	T/F	CW	N	N	N	Bottom surface very smooth, no residue.	Multiple cracks and voids along length of tube. Tiny voids apparent in un-used sections of tube.	Y	Y	N	Fracture horizontal crack growth on top cheek edge, growth outer bore to inner bore, from collection of smaller voids	6.4	2.4	2.66	1.48
[illegible]	Discharge end, inner and outer edge.(Sites also evident suction end).	193	2316000	FW5	40°C	100rpm	T/F	CW	N	N	N	Bottom surface very smooth, no residue.	Multiple cracks and voids along length of tube. Tiny voids apparent in un-used sections of tube.	Y	Y	N	Fracture horizontal crack growth on top cheek edge, growth outer bore to inner bore, from collection of smaller voids	6.4	2.4	2.66	1.45
[illegible]	Discharge end, inner and outer edge.	188	2256000	FW5	40°C	100rpm	T/F	CW	N	N	N	Bottom surface very smooth, no residue.	Multiple cracks and voids along length of tube. Tiny voids apparent in un-used sections of tube.	Y	Y	N	Fracture horizontal crack growth on top cheek edge, growth outer bore to inner bore, from collection of smaller voids	6.4	2.4	2.66	1.44
2332	Discharge end, inner and outer edge.	184	2208000	FW5	4°C	100rpm	T/F	CW	N	N	N	Bottom of tube showing silicone discharge.Top surface of tube showing residue and roughening thro' micro cracks.	Multiple cracks and voids along length of tube. Tiny voids apparent in un-used sections of tube.	Y	Y	N	Fractures - two types, vertical fracture traversing cheek inner and outer edge. Other fracture type on bottom cheek edge.	9.6	2.4	4	1.55
2341	Between mid-sweep & discharge end, inner and outer edge.	226	2712000	FW5	4°C	100rpm	T/F	CW	Bottom surface of tube displaying broad chevrons	N	Outer edge cheek showing deep scalloping, does not seem to have contributed ti fracture though.		Multiple cracks and voids along length of tube. Tiny voids apparent in un-used sections of tube.	N	N	N	Fractures as 2332.Crack growth is vertical from inner to outer cheek.Also showing crack growth on bottom surface, on inner bore, mid sweep, not broken thro to outer bore.	9.6	2.4	4	1.49
2347	Discharge end, inner and outer edge.	389	4668000	FW5	4°C	100rpm	T/F	CW	N	N	Deep scalloping of inner edge cheek, not contributed to fracture.		Multiple cracks and voids along length of tube. Tiny voids apparent in un-used sections of tube.	N	N	Y	Crack growth on bottom surface on inner bore, as described for 2341. Fracture type vertical growth traversing top and bottom cheek	9.6	2.4	4	1.54
2376	Discharge end, inner and outer edge.	356	4272000	FW5	4°C	100rpm	T/F	CW	N	N	Scalloping both on inner and outer edge on top of cheek.	Shallow chevrons apparent on bottom surface along with crack growth on inner bore as per 2341	Multiple cracks and voids along length of tube. Tiny voids apparent in un-used sections of tube.	Y	N	Y	Fracture vertid fracture traversing cheek.	9.6	2.4	4	1.66

9.9 Appendix 9: Swell Measurement - variation with time

	Hours after first immersion that measurement was taken							
Database Number	**Start weight**	**3.2**	**22.7**	**48**	**72**	**96**		**Vol Swell**
2303	1.34	3	3.15	3.22	3.22	3.22		1.402985
2360	1.27	2.87	3.03	3.07	3.07	3.07		1.417323
2338	1.25	2.84	3.01	3.06	3.06	3.06		1.448
2303	0.25	0.61	0.61	0.63	0.63	0.63		1.52
2360	0.26	0.63	0.65	0.64	0.64	0.64		1.461538
2338	0.34	0.81	0.83	0.84	0.84	0.84		1.470588

9.10 Appendix 10: Swell Measurement – variation with geometry

Geometry No	Database Number	Sample No	Pre Swell weight (g)	Post Swell weight (g)	Swell Ratio
1	2387	1	0.56	1.38	1.46
2	2387	2	0.52	1.24	1.38
3	2387	3	0.47	1.12	1.38
4	2387	4	0.47	1.14	1.43
5	2387	5	0.52	1.28	1.46
6	2387	6	0.58	1.43	1.47
7	2387	7	0.44	1.12	1.55
8	2387	8	0.49	1.25	1.55
1	2399	1	0.49	1.19	1.43
2	2399	2	0.49	1.29	1.63
3	2399	3	0.44	1.16	1.64
4	2399	4	0.48	1.24	1.58
5	2399	5	0.49	1.2	1.45
6	2399	6	0.47	1.16	1.47
7	2399	7	0.48	1.28	1.67
8	2399	8	0.48	1.24	1.58
9	2399	9	0.47	1.21	1.57

9.11 Appendix 11: Swell Measurement – weighing method accuracy

	Pre Swell Tolerance Analysis			Post Swell Tolerance Analysis			
EXP 14	**MAX**	**MIN**	**Range**	**MAX**	**MIN**	**Average**	**Range**
Extruded1	0.44	0.42	0.02	1.08	1.05	1.055	0.05
Extruded2	0.43	0.42	0.01	1.1	1.07	1.085	0.03
Extruded3	0.47	0.45	0.02	1.18	1.14	1.156	0.04
Extruded4	0.49	0.47	0.02	1.22	1.18	1.204	0.04
Extruded5	0.45	0.42	0.03	1.1	1.05	1.073	0.05
Extruded6	0.43	0.42	0.01	1.08	1.05	1.066	0.03
Extruded7	0.47	0.46	0.02	1.18	1.14	1.166	0.04
Extruded8	0.39	0.38	0.01	0.99	0.95	0.966	0.04
Extruded9	0.42	0.39	0.03	1.06	1.02	1.043	0.04

9.12 Appendix 12: Hoop stress calculations for tube sizes used

	mm	mm	mm	mm	mm	Pressure in N/mm²							
Size	**Bore**	**a²**	**b²**	**r²**	**WT**	**0.05**	**0.10**	**0.15**	**0.20**	**0.25**	**0.30**	**0.40**	**0.70**
0.5 x 1.6	0.5	0.0625	13.69	0.0625	1.6	0.05	0.09	0.14	0.19	0.24	0.28	0.38	0.66
0.8 x 1.6	0.8	0.16	16	0.16	1.6	0.04	0.09	0.13	0.17	0.21	0.26	0.34	0.60
1.6 x 1.6	1.6	0.64	23.04	0.64	1.6	0.02	0.04	0.06	0.08	0.10	0.12	0.16	0.27
1.6 x 2.4	1.6	0.64	40.96	0.64	2.4	0.02	0.04	0.06	0.08	0.09	0.11	0.15	0.26
3.2 x 1.6	3.2	2.56	40.96	2.56	1.6	-0.07	-0.15	-0.22	-0.30	-0.37	-0.45	-0.60	-1.05
3.2 x 1.8	3.2	2.56	46.24	2.56	1.8	-0.08	-0.15	-0.23	-0.30	-0.38	-0.45	-0.60	-1.05
3.2 x 2.4	3.2	2.56	64	2.56	2.4	-0.08	-0.15	-0.23	-0.30	-0.38	-0.46	-0.61	-1.06
4.8 x 1.6	4.8	5.76	64	5.76	1.6	-0.23	-0.47	-0.70	-0.93	-1.17	-1.40	-1.87	-3.27
4.8 x 2.4	4.8	5.76	92.16	5.76	2.4	-0.23	-0.47	-0.70	-0.94	-1.17	-1.41	-1.88	-3.29
6 x 2.1	6	9	104.04	9	2.1	-0.40	-0.79	-1.19	-1.58	-1.98	-2.37	-3.17	-5.54
6.4 x 1.6	6.4	10.24	92.16	10.24	1.6	-0.46	-0.91	-1.37	-1.83	-2.28	-2.74	-3.65	-6.39
6.4 x 2.4	6.4	10.24	125.44	10.24	2.4	-0.46	-0.92	-1.37	-1.83	-2.29	-2.75	-3.66	-6.41
8.0 x 1.6	8	16	125.44	16	1.6	-0.74	-1.49	-2.23	-2.97	-3.72	-4.46	-5.95	-10.41
8.0 x 2.4	8	16	163.84	16	2.4	-0.75	-1.49	-2.24	-2.98	-3.73	-4.47	-5.96	-10.43
9.6 x 2.4	9.6	23.04	207.36	23.04	2.4	-1.10	-2.19	-3.29	-4.39	-5.48	-6.58	-8.77	-15.35
9.6 x 3.2	9.6	23.04	256	23.04	3.2	-1.10	-2.20	-3.29	-4.39	-5.49	-6.59	-8.78	-15.37
9.6 x 4.8	9.6	23.04	368.64	23.04	4.8	-1.10	-2.20	-3.30	-4.40	-5.49	-6.59	-8.79	-15.38
12.7 x 3.2	12.7	40.3225	364.81	40.3225	3.2	-1.96	-3.92	-5.88	-7.84	-9.80	-11.76	-15.68	-27.45
12.7 x 4.8	12.7	40.3225	497.29	40.3225	4.8	-1.96	-3.92	-5.89	-7.85	-9.81	-11.77	-15.70	-27.47
12 x 4	12	36	400	36	4	-1.75	-3.49	-5.24	-6.98	-8.73	-10.47	-13.96	-24.44
15.9 x 3.2	15.9	63.2025	497.29	63.2025	3.2	-3.10	-6.21	-9.31	-12.42	-15.52	-18.62	-24.83	-43.45
15.9 x 4.8	15.9	63.2025	650.25	63.2025	4.8	-3.11	-6.21	-9.32	-12.42	-15.53	-18.63	-24.84	-43.47
16 x 4	16	64	576	64	4	-3.14	-6.29	-9.43	-12.58	-15.72	-18.87	-25.16	-44.02
19 x 4.8	19	90.25	817.96	90.25	4.8	-4.46	-8.91	-13.37	-17.83	-22.28	-26.74	-35.66	-62.40
25.4 x 4.8	25.4	161.29	1225	161.29	4.8	-8.01	-16.02	-24.02	-32.03	-40.04	-48.05	-64.06	-112.11

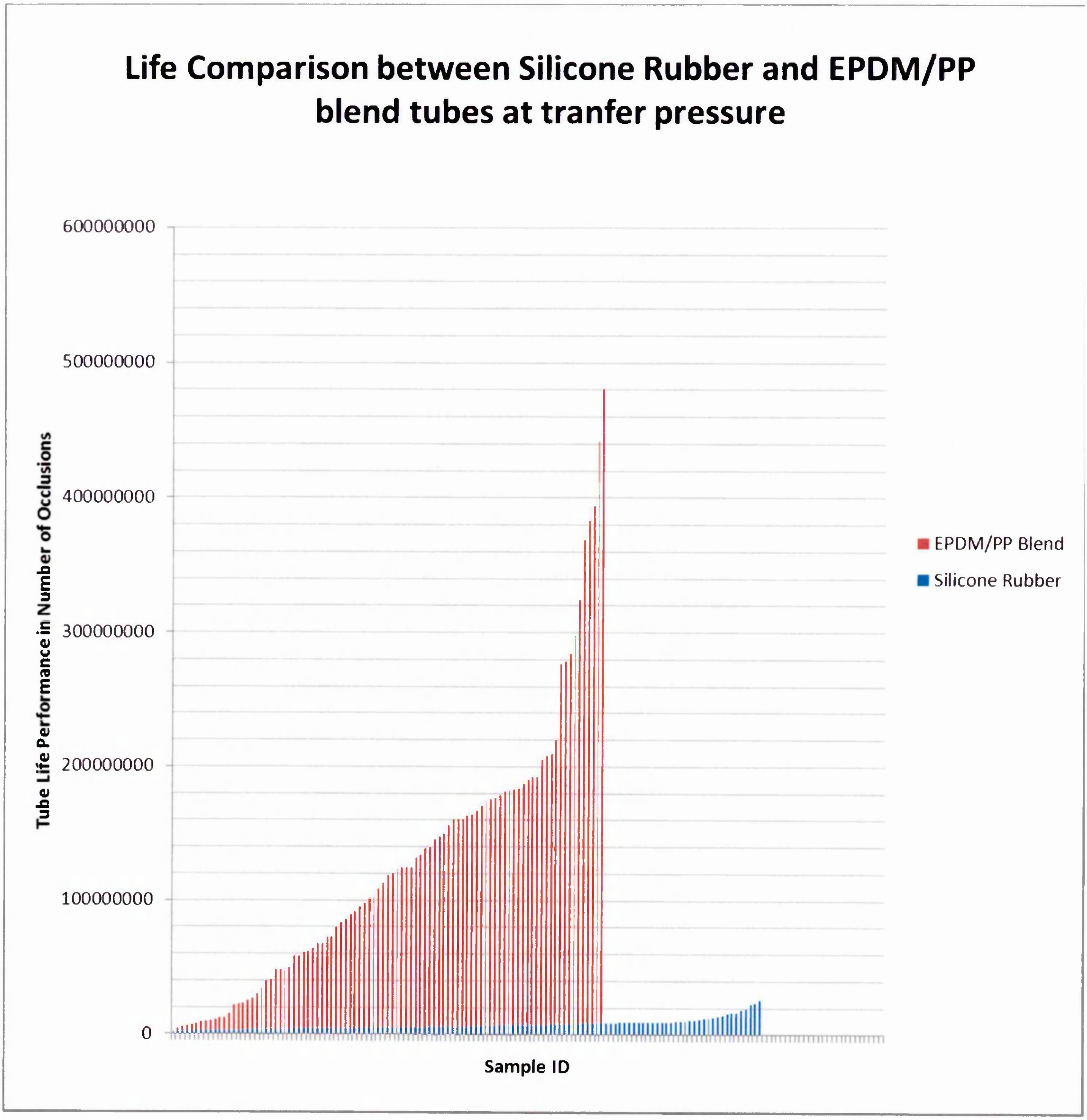
9.13 Appendix 13: Volume and surface area calculations for tube sizes used

Size	Bore	WT	R	r	Surface Area	Volume
0.5 x 1.6	0.5	1.6	1.85	0.25	34.31	10.56
0.8 x 1.6	0.8	1.6	2	0.4	39.21	12.06
1.6 x 1.6	1.6	1.6	2.4	0.8	52.28	16.08
1.6 x 2.4	1.6	2.4	3.2	0.8	85.45	30.16
3.2 x 1.6	3.2	1.6	3.2	1.6	78.41	24.13
3.2 x 1.8	3.2	1.8	3.4	1.6	87.96	28.27
3.2 x 2.4	3.2	2.4	4	1.6	119.63	42.22
4.8 x 1.6	4.8	1.6	4	2.4	104.55	32.17
4.8 x 2.4	4.8	2.4	4.8	2.4	153.81	54.29
6 x 2.1	6	2.1	5.1	3	157.77	53.44
6.4 x 1.6	6.4	1.6	4.8	3.2	130.69	40.21
6.4 x 2.4	6.4	2.4	5.6	3.2	187.99	66.35
8.0 x 1.6	8	1.6	5.6	4	156.83	48.25
8.0 x 2.4	8	2.4	6.4	4	222.17	78.41
9.6 x 2.4	9.6	2.4	7.2	4.8	256.35	90.48
9.6 x 3.2	9.6	3.2	8	4.8	337.78	128.68
9.6 x 4.8	9.6	4.8	9.6	4.8	524.77	217.15
12.7 x 3.2	12.7	3.2	9.55	6.35	419.59	159.84
12.7 x 4.8	12.7	4.8	11.15	6.35	637.74	263.89
12 x 4	12	4	10	6	502.65	201.06
15.9 x 3.2	15.9	3.2	11.15	7.95	504.04	192.01
15.9 x 4.8	15.9	4.8	12.75	7.95	754.36	312.15
16 x 4	16	4	12	8	628.32	251.33
19 x 4.8	19	4.8	14.3	9.5	867.33	358.90
25.4 x 4.8	25.4	4.8	17.5	12.7	1100.56	455.41

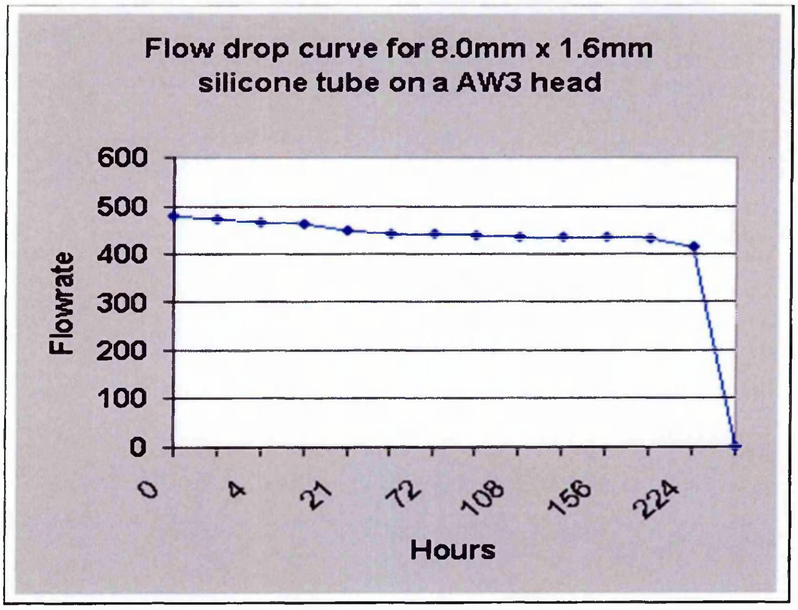
9.14 Appendix 14: Aspect Ratio calculations for tube sizes used

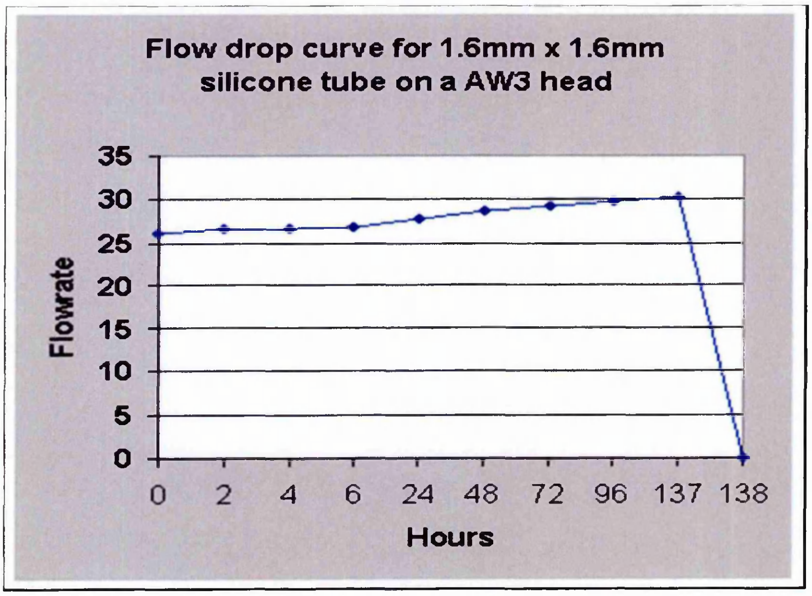
Size	Bore	WT	Aspect ratio
0.5 x 1.6	0.5	1.6	0.31
0.8 x 1.6	0.8	1.6	0.50
1.6 x 1.6	1.6	1.6	1.00
1.6 x 2.4	1.6	2.4	0.67
3.2 x 1.6	3.2	1.6	2.00
3.2 x 1.8	3.2	1.8	1.78
3.2 x 2.4	3.2	2.4	1.33
4.8 x 1.6	4.8	1.6	3.00
4.8 x 2.4	4.8	2.4	2.00
6 x 2.1	6	2.1	2.86
6.4 x 1.6	6.4	1.6	4.00
6.4 x 2.4	6.4	2.4	2.67
8.0 x 1.6	8	1.6	5.00
8.0 x 2.4	8	2.4	3.33
9.6 x 2.4	9.6	2.4	4.00
9.6 x 3.2	9.6	3.2	3.00
9.6 x 4.8	9.6	4.8	2.00
12.7 x 3.2	12.7	3.2	3.97
12.7 x 4.8	12.7	4.8	2.65
12 x 4	12	4	3.00
15.9 x 3.2	15.9	3.2	4.97
15.9 x 4.8	15.9	4.8	3.31
16 x 4	16	4	4.00
19 x 4.8	19	4.8	3.96
25.4 x 4.8	25.4	4.8	5.29

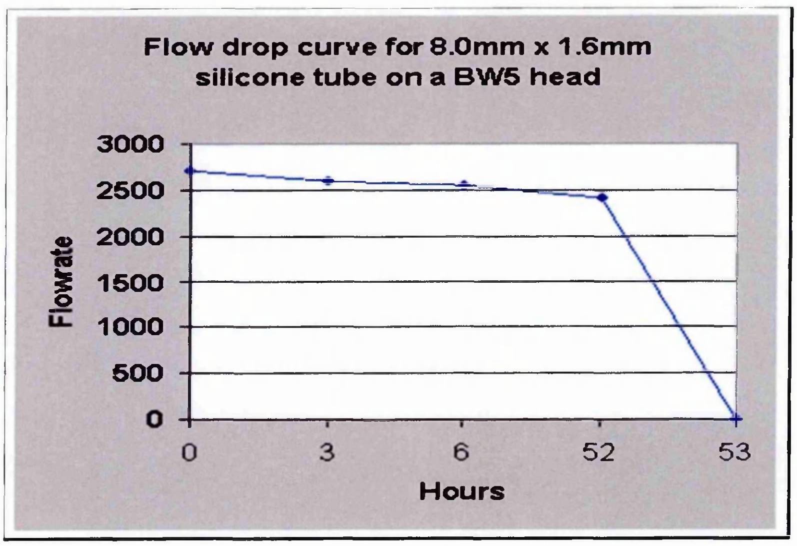
9.15 Appendix 15: Silicone life distribution with EPDM/PP life distribution for historical data held

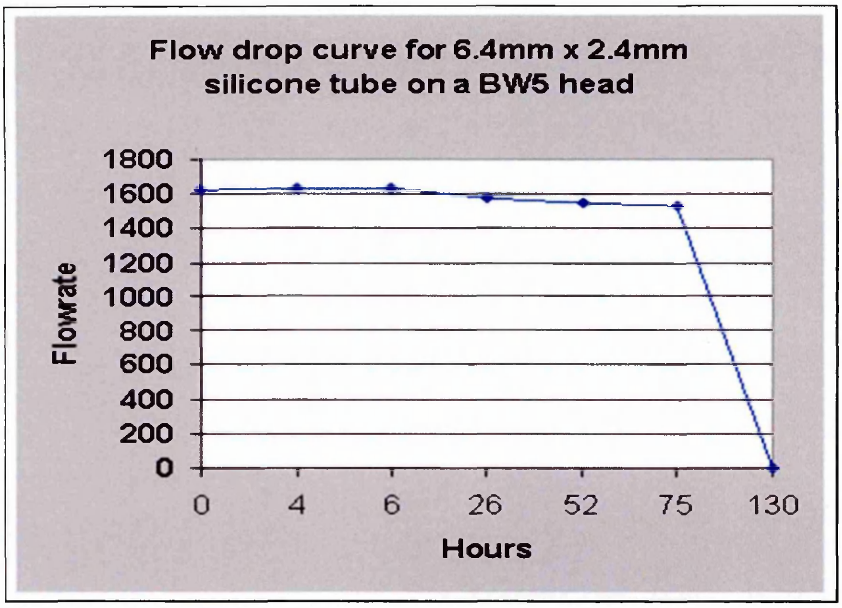


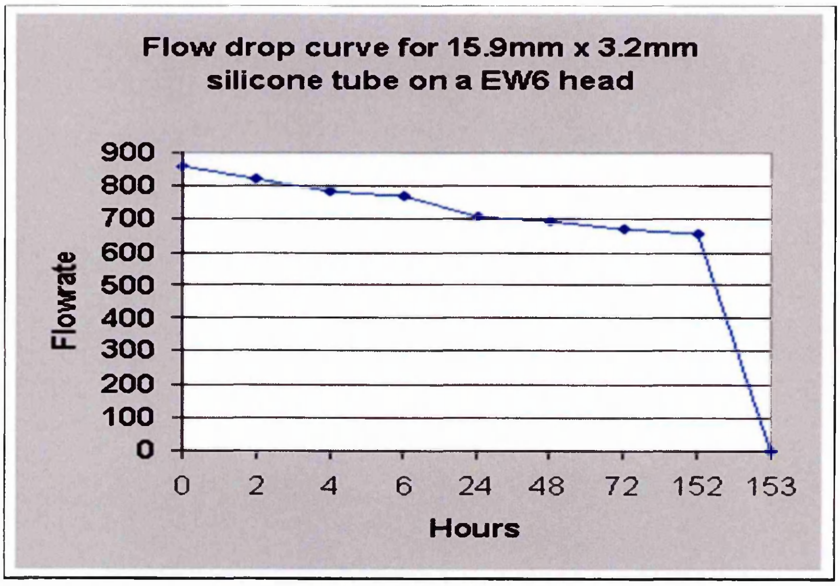
9.16 Appendix 16: Flow profiles for different pump head types across silicone tube sizes

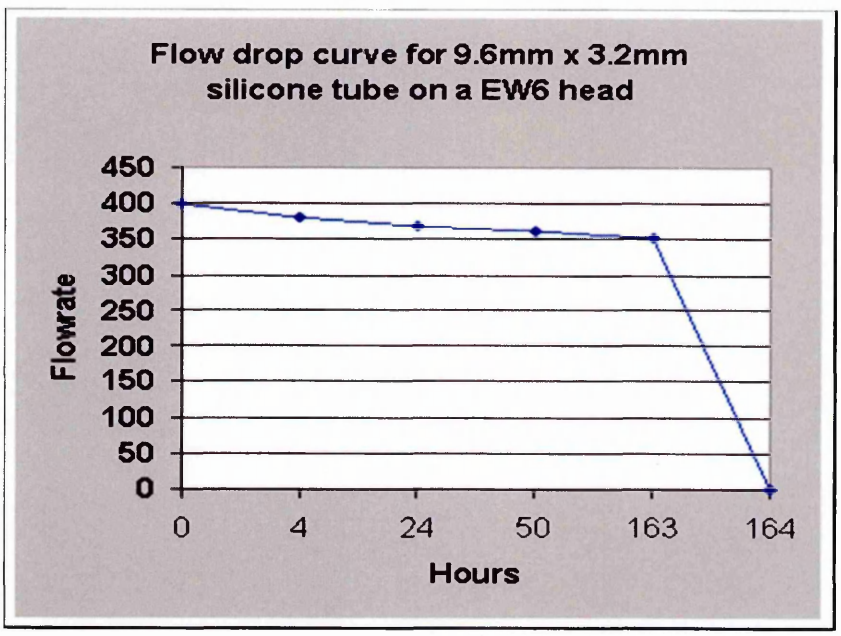


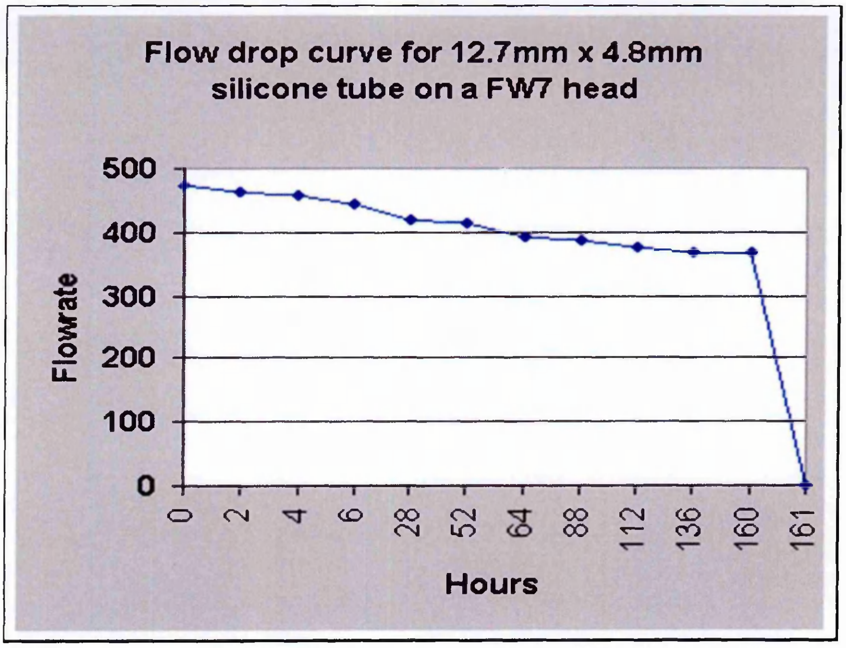


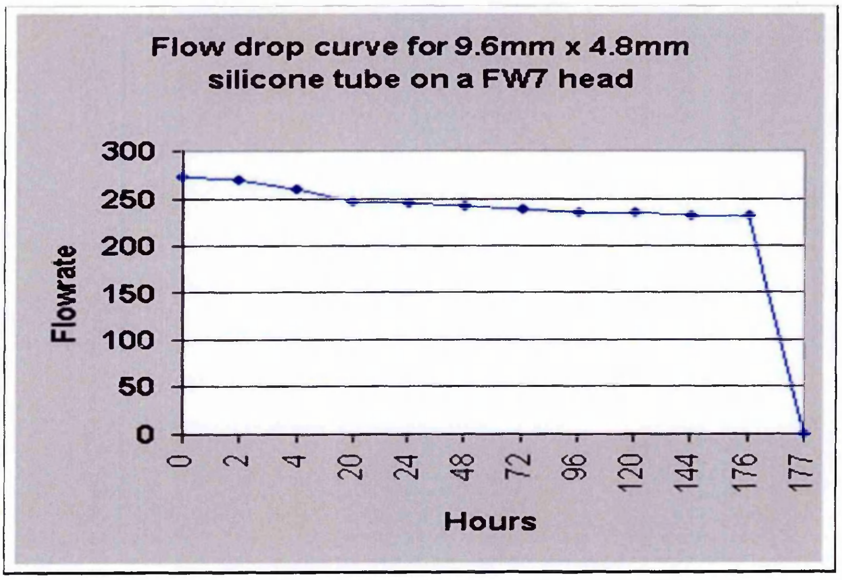


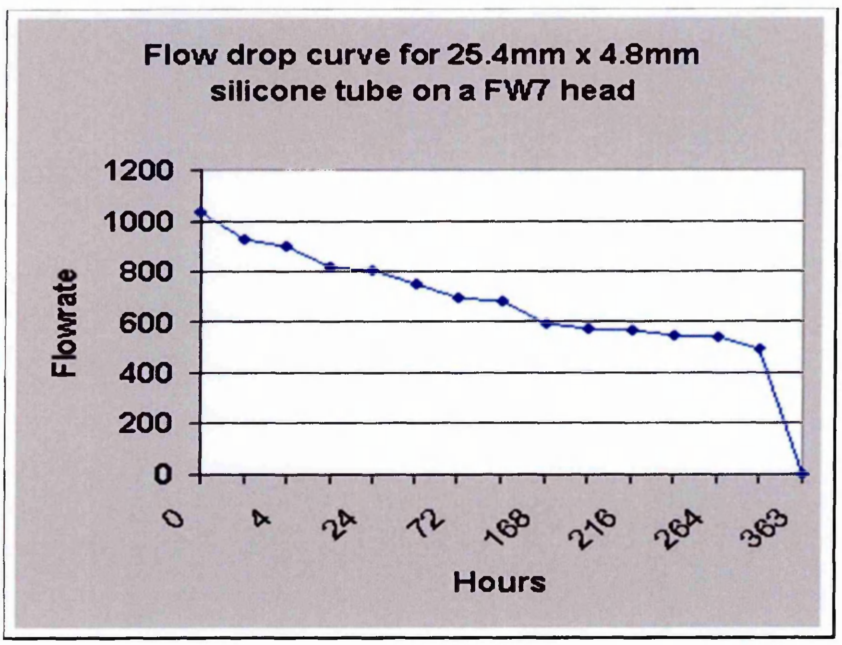

Flow drop curve for 6.4mm x 2.4mm
silicone tube on a BW5 head
Flowrate
1800
1600
1400
1200
1000
800
600
400
200
0
0
4
6
26
52
75
130
Hours


Flow drop curve for 15.9mm x 3.2mm
silicone tube on a EW6 head
Flowrate
900
800
700
600
500
400
300
200
100
0
0
2
4
6
24
48
72
152
153
Hours


Flow drop curve for 9.6mm x 3.2mm
silicone tube on a EW6 head
Flowrate
450
400
350
300
250
200
150
100
50
0
0
4
24
50
163
164
Hours


Flow drop curve for 12.7mm x 4.8mm silicone tube on a FW7 head
Flowrate
500
400
300
200
100
0
0
2
4
6
28
52
64
88
112
136
160
161
Hours

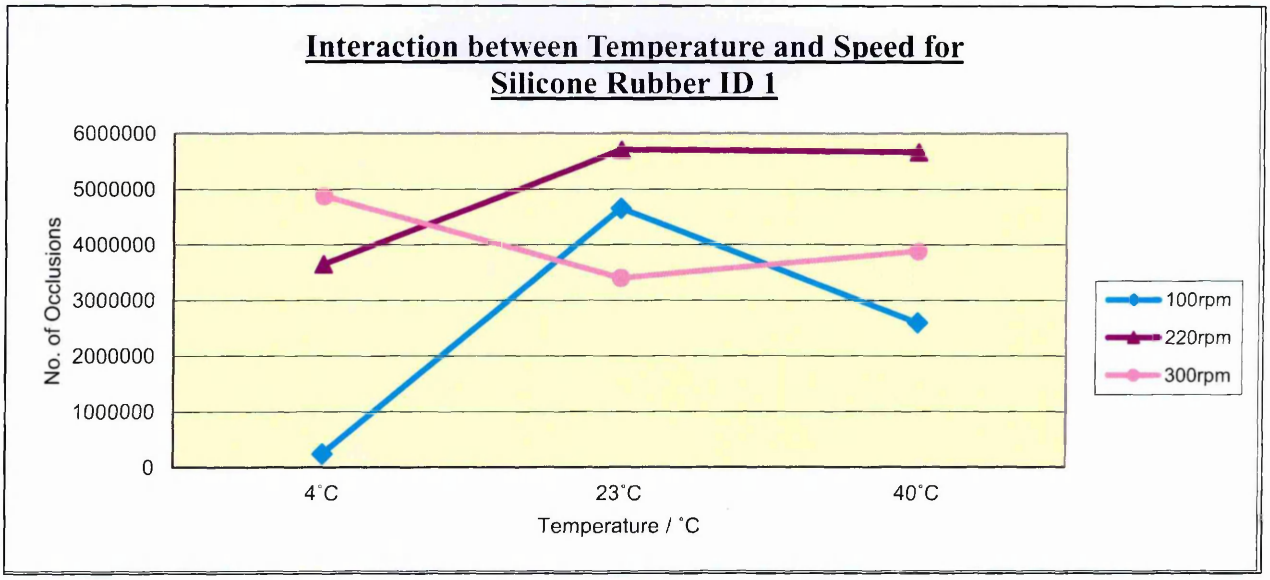

Flow drop curve for 9.6mm x 4.8mm silicone tube on a FW7 head
Flowrate
300
250
200
150
100
50
0
0
2
4
20
24
48
72
96
120
144
176
177
Hours


Flow drop curve for 25.4mm x 4.8mm silicone tube on a FW7 head
Flowrate
1200
1000
800
600
400
200
0
0
4
24
72
168
216
264
363
Hours

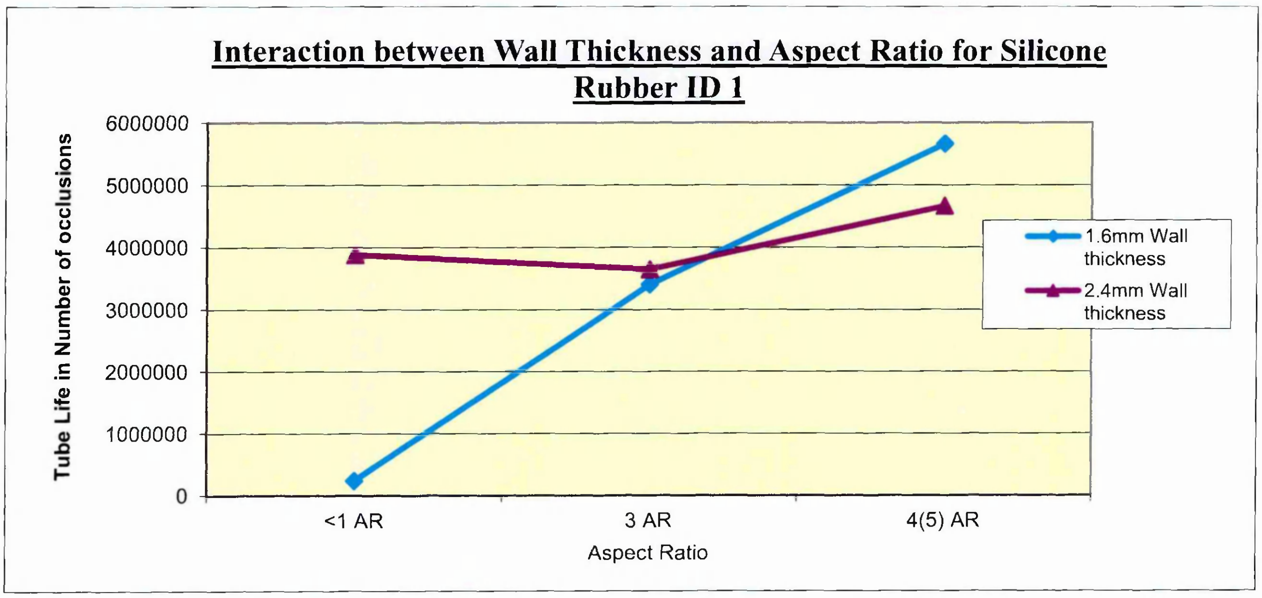
9.17 Appendix 17: Interaction between factors – Silicone Material 1

DOE Matrix Design:

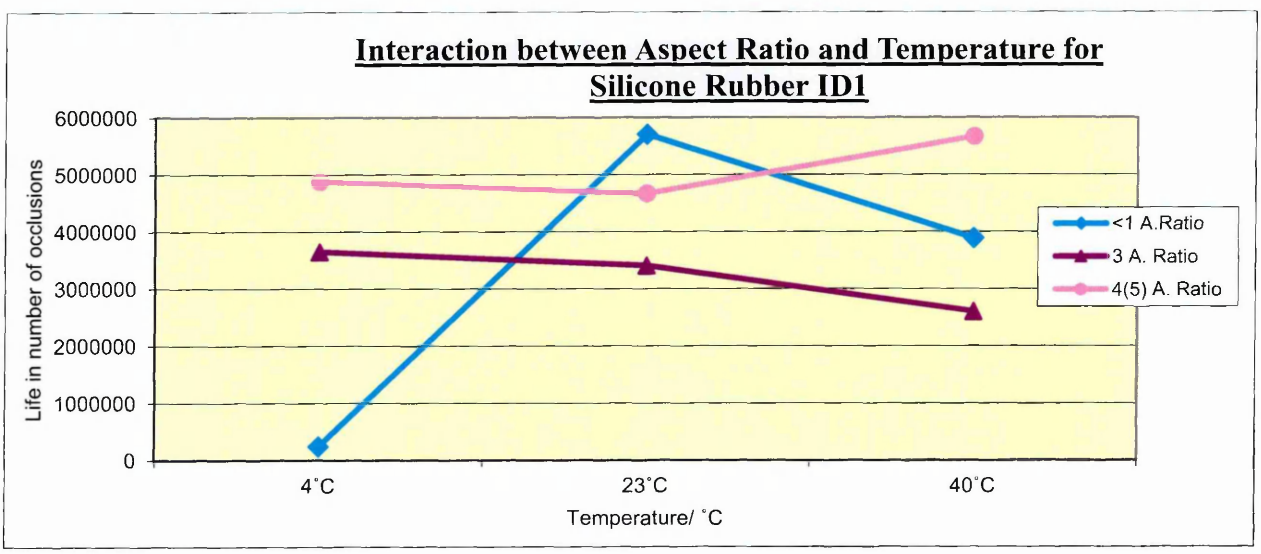
Factor		1 A Temp °C		2 B Wall T mm		3 C Asp. R		4 D Speed rpm	
Level	3	40		2.4		4(5)		300	
	2	23		2.4		3		220	Analysis
Run	1	4		1.6		<1		100	No
1	1	4	2	2.4	2	3	2	220	2
2	1	4	3	2.4	3	4	3	300	3
3	2	23	2	2.4	3	4	1	100	5
4	3	40	2	2.4	1	<1	3	300	8
5	3	40	3	2.4	2	3	1	100	9
6	1	4	1	1.6	1	<1	1	100	1
7	2	23	3	2.4	1	<1	2	220	6
8	2	23	1	1.6	2	3	3	300	4
9	3	40	1	1.6	3	4	2	220	7



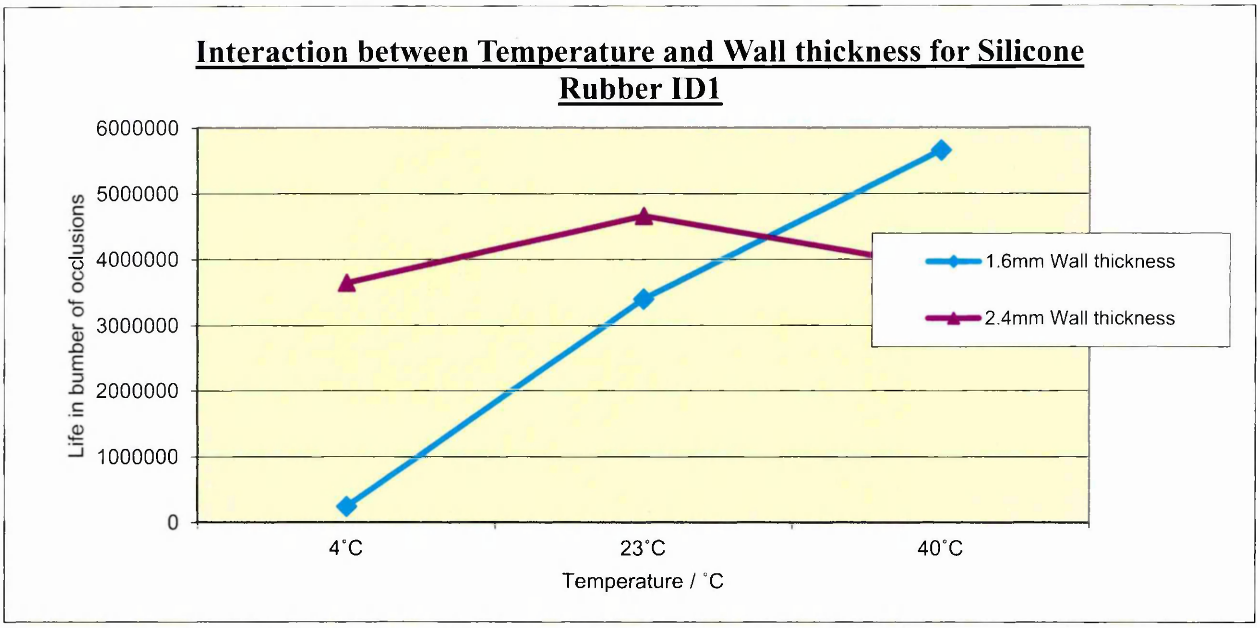
Interaction between Temperature and Speed for Silicone Rubber ID 1
6000000
5000000
4000000
3000000
2000000
1000000
0
No. of Occlusions
4˚C
23˚C
40˚C
Temperature / ˚C
100rpm
220rpm
300rpm

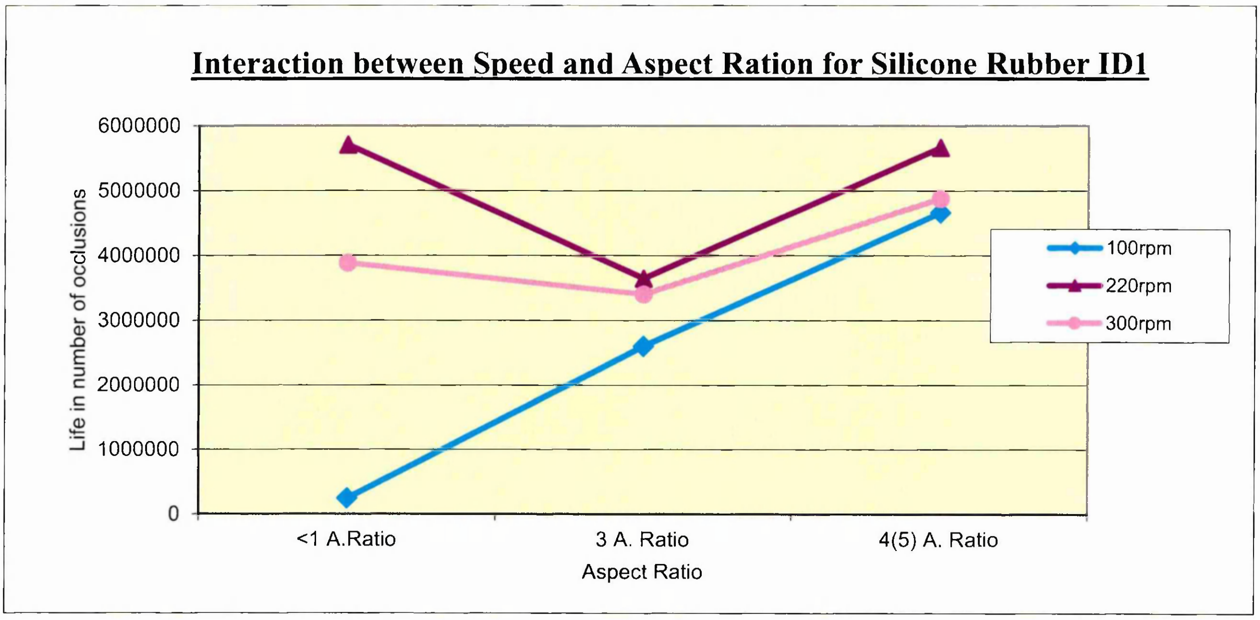


Interaction between Wall Thickness and Aspect Ratio for Silicone Rubber ID 1
6000000
5000000
4000000
3000000
2000000
1000000
0
Tube Life in Number of occlusions
<1 AR
3 AR
4(5) AR
Aspect Ratio
1.6mm Wall thickness
2.4mm Wall thickness

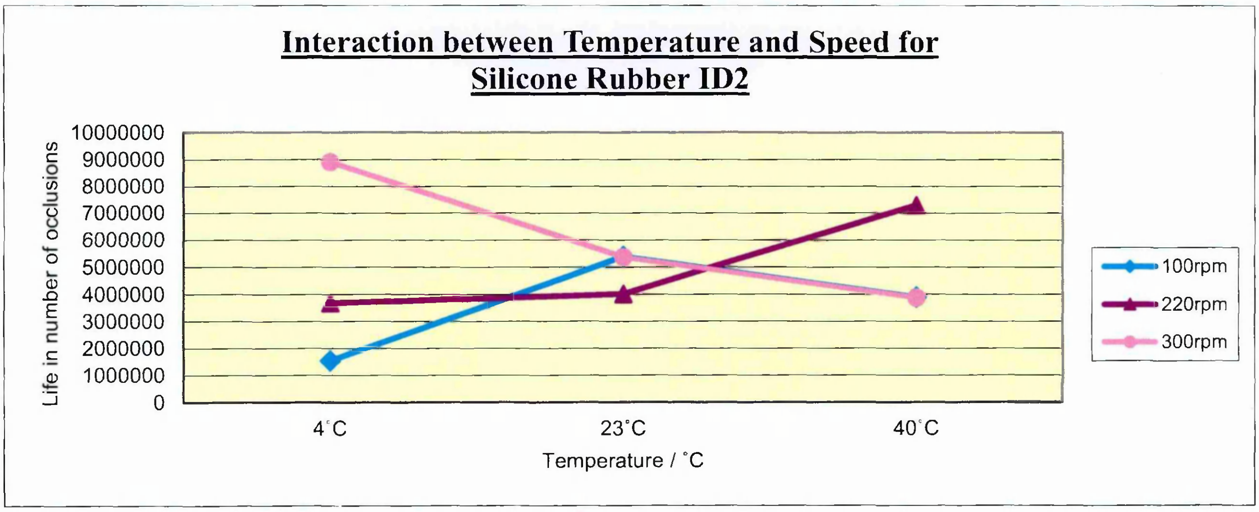


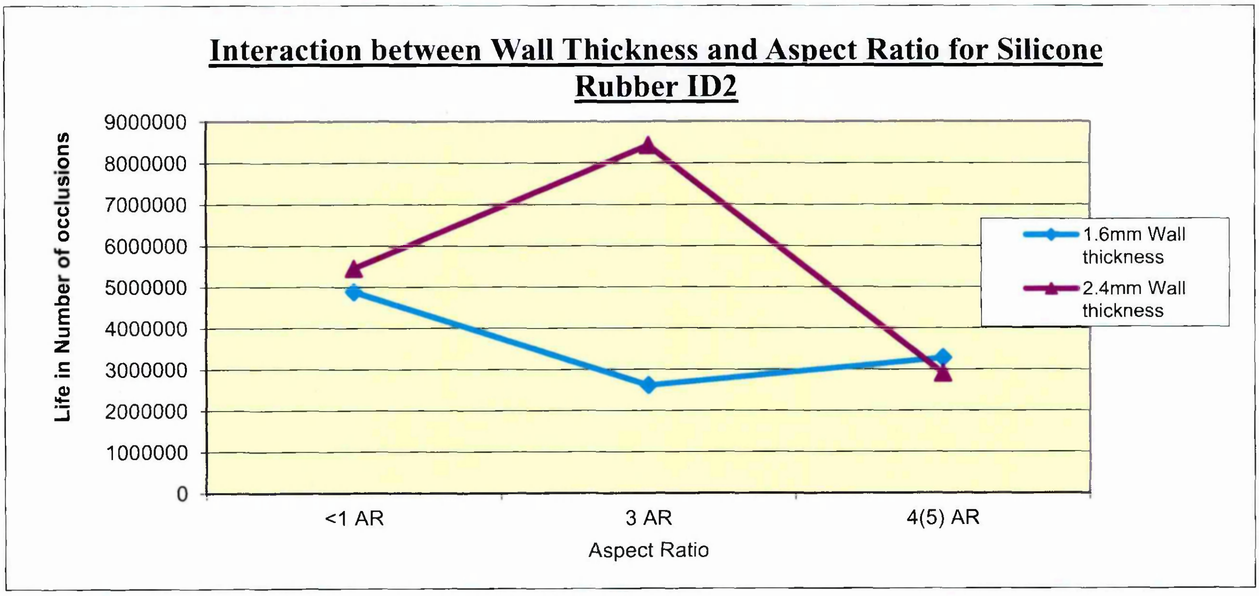
Interaction between Aspect Ratio and Temperature for Silicone Rubber ID1
6000000
5000000
4000000
3000000
2000000
1000000
0
Life in number of occlusions
4˚C
23˚C
40˚C
Temperature/ ˚C
<1 A.Ratio
3 A. Ratio
4(5) A. Ratio

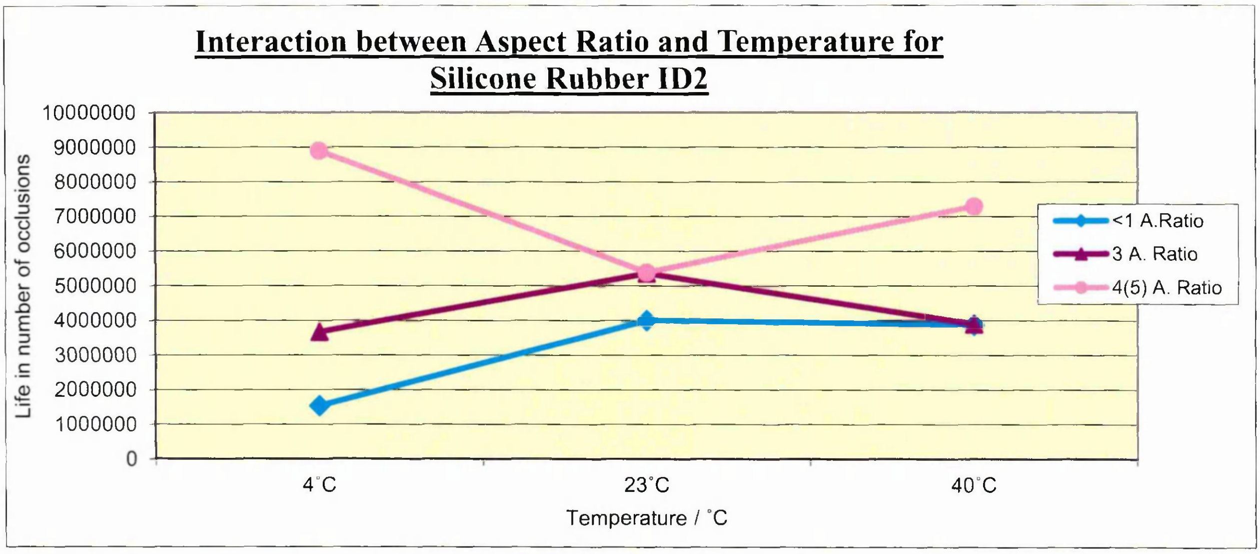

Interaction between Temperature and Wall thickness for Silicone Rubber ID1
6000000
5000000
4000000
3000000
2000000
1000000
0
Life in bumber of occlusions
4°C
23°C
40°C
Temperature / °C
1.6mm Wall thickness
2.4mm Wall thickness

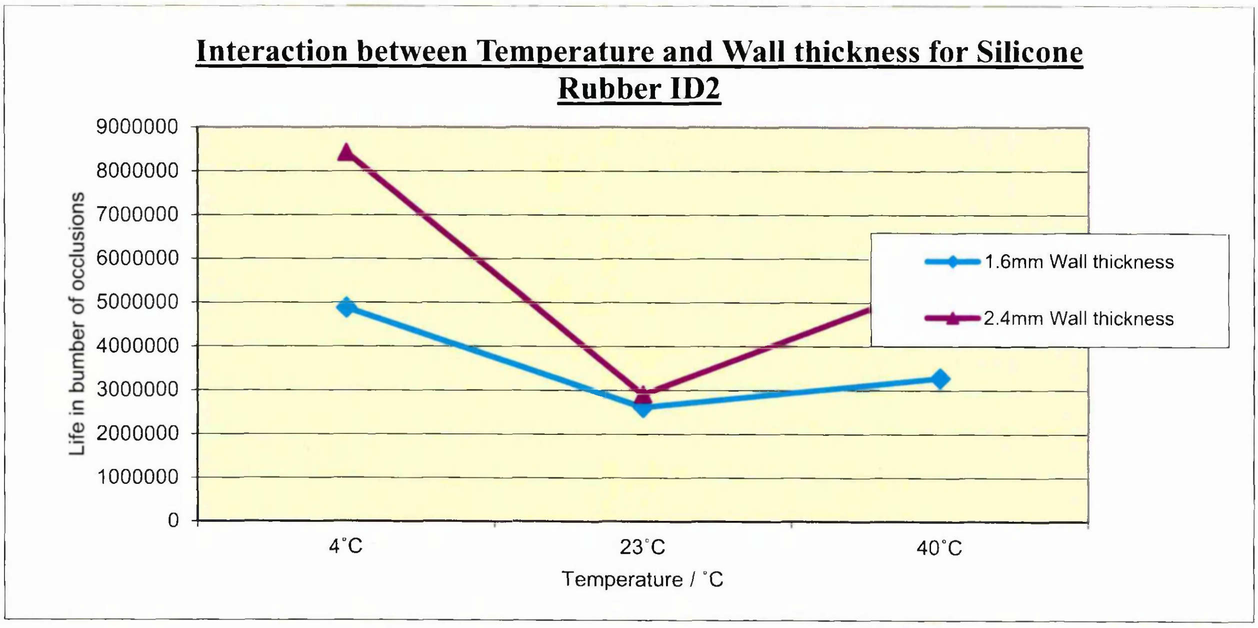

Interaction between Speed and Aspect Ration for Silicone Rubber ID1
6000000
5000000
4000000
3000000
2000000
1000000
0
Life in number of occlusions
<1 A.Ratio
3 A. Ratio
4(5) A. Ratio
Aspect Ratio
100rpm
220rpm
300rpm

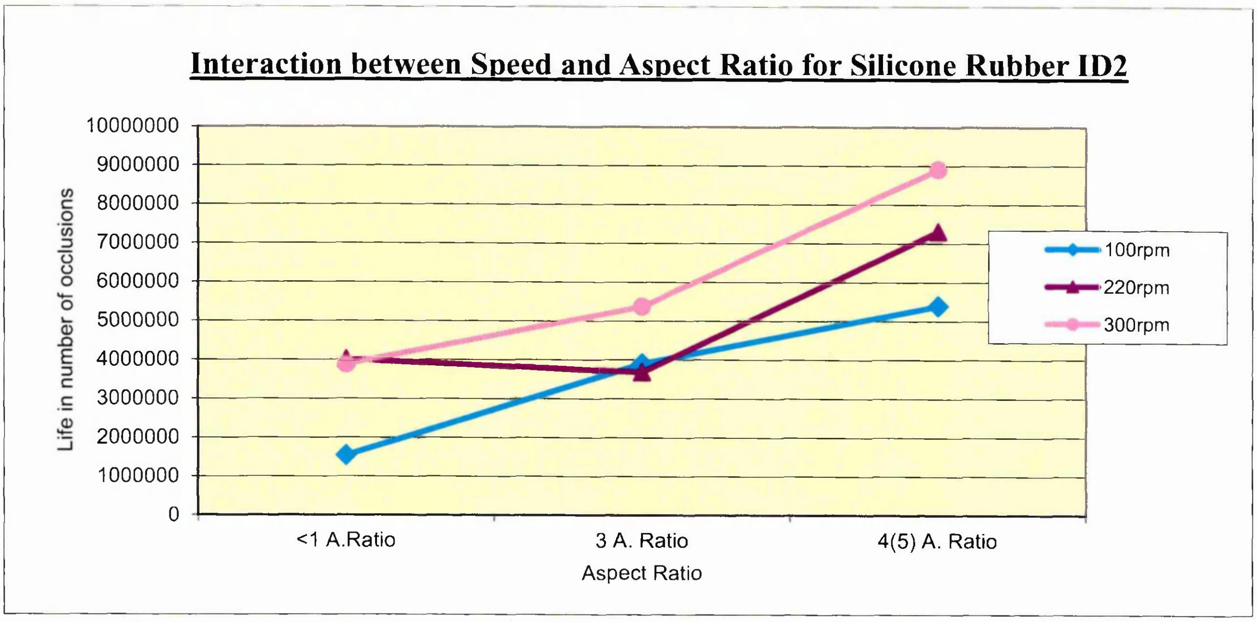
9.18 Appendix 18: Interaction between factors – Silicone Material 2



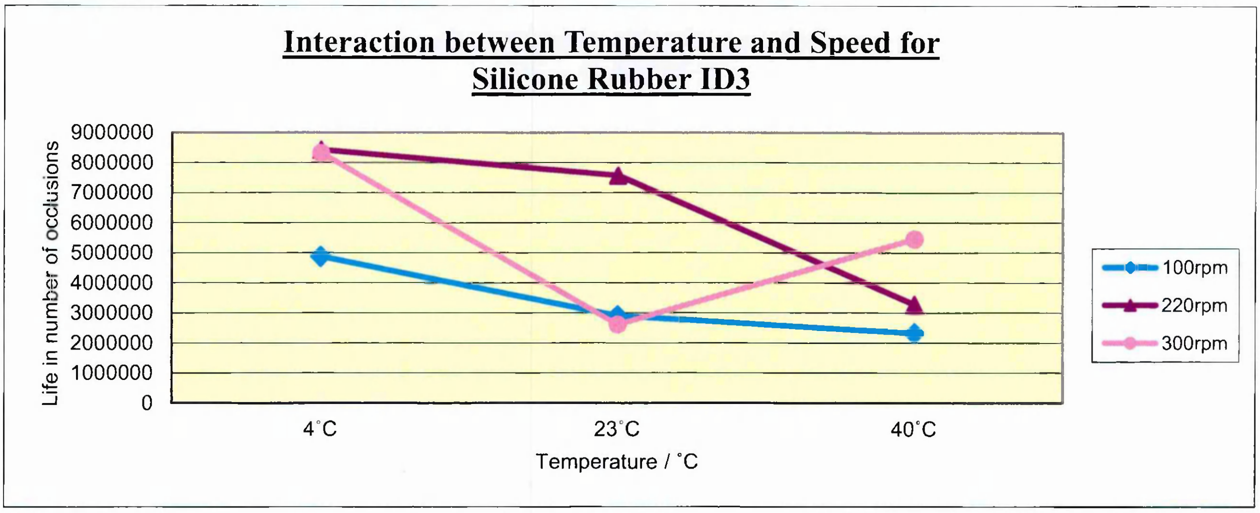


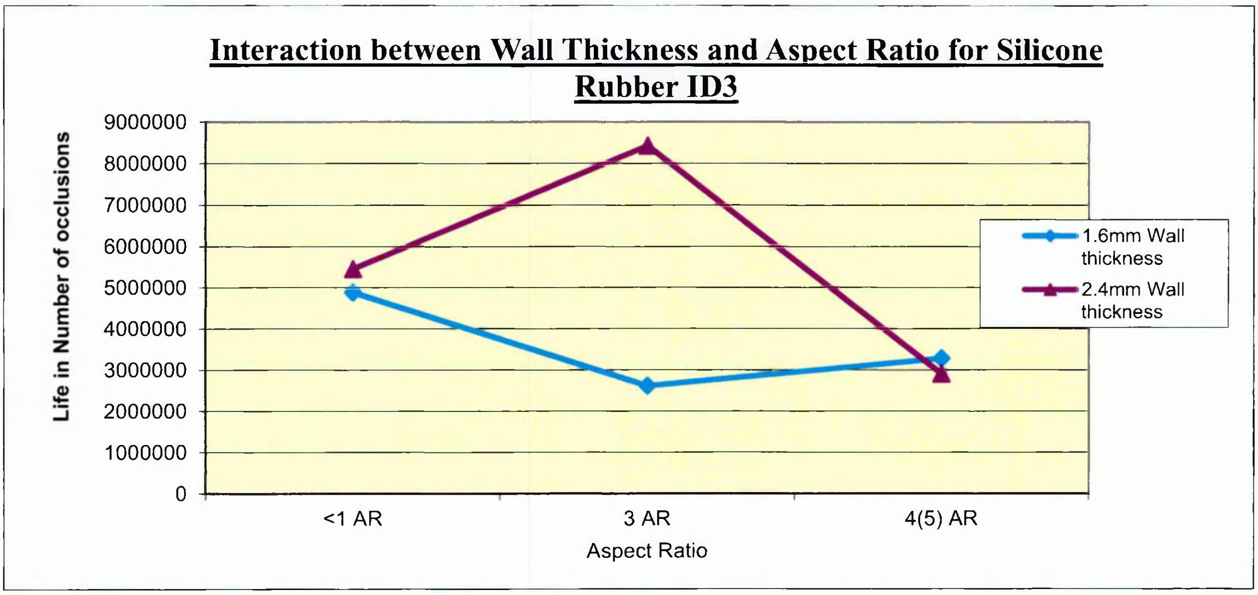

Interaction between Aspect Ratio and Temperature for Silicone Rubber ID2
10000000
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
Life in number of occlusions
4°C
23°C
40°C
Temperature / °C
<1 A.Ratio
3 A. Ratio
4(5) A. Ratio

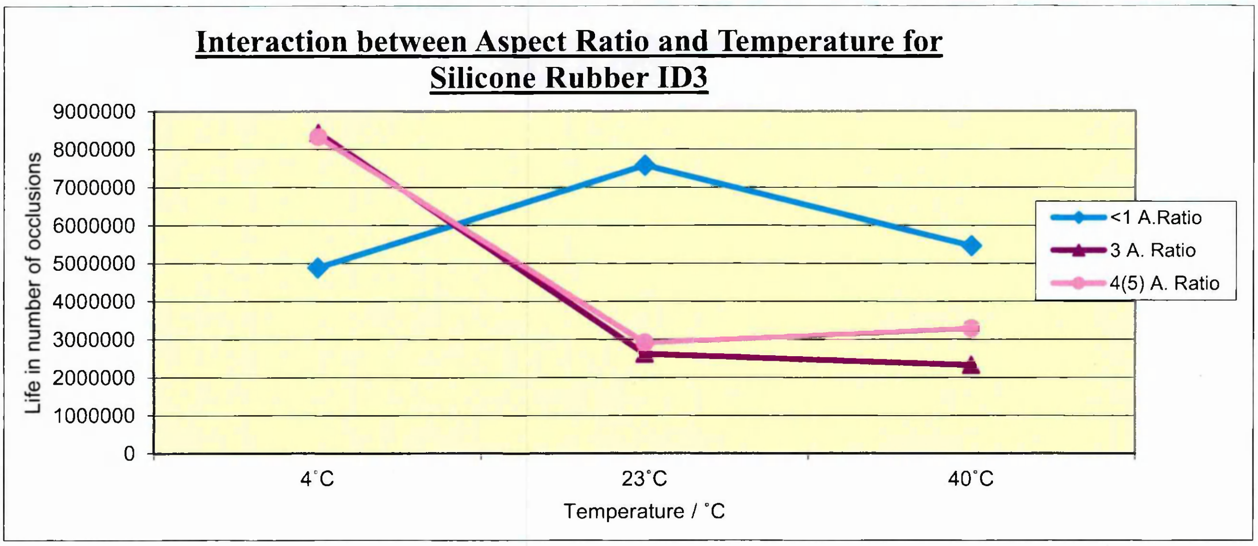

Interaction between Temperature and Wall thickness for Silicone Rubber ID2
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
Life in bumber of occlusions
4°C
23°C
40°C
Temperature / °C
1.6mm Wall thickness
2.4mm Wall thickness

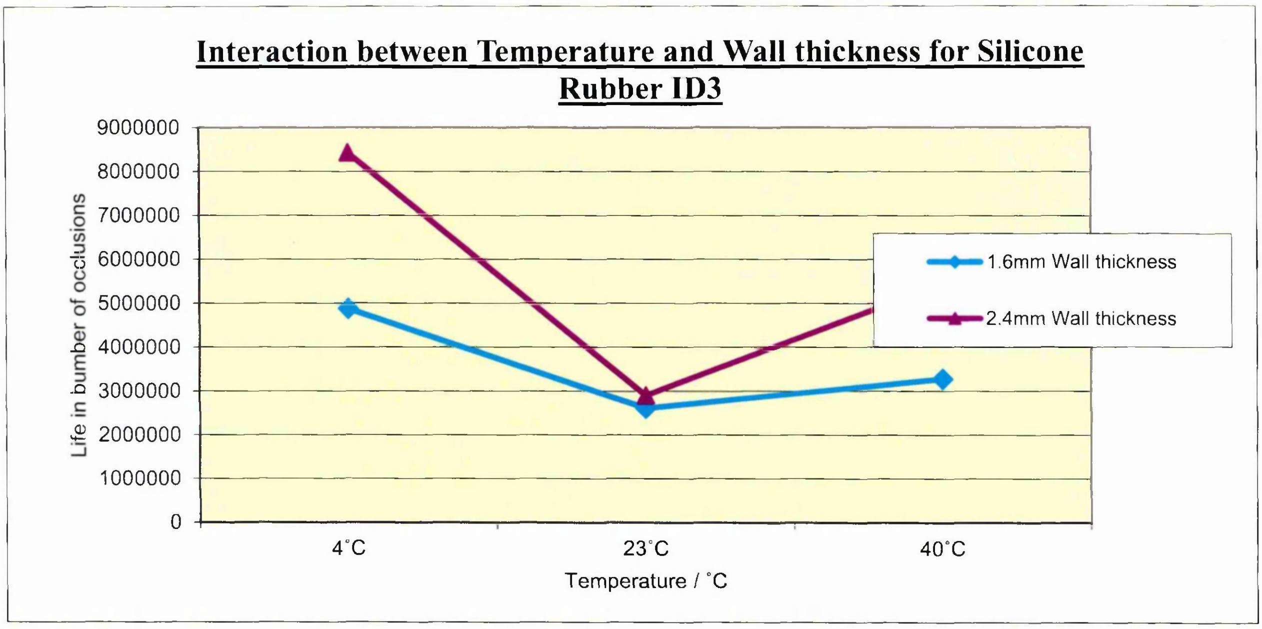

Interaction between Speed and Aspect Ratio for Silicone Rubber ID2
10000000
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
Life in number of occlusions
<1 A.Ratio
3 A. Ratio
4(5) A. Ratio
Aspect Ratio
100rpm
220rpm
300rpm

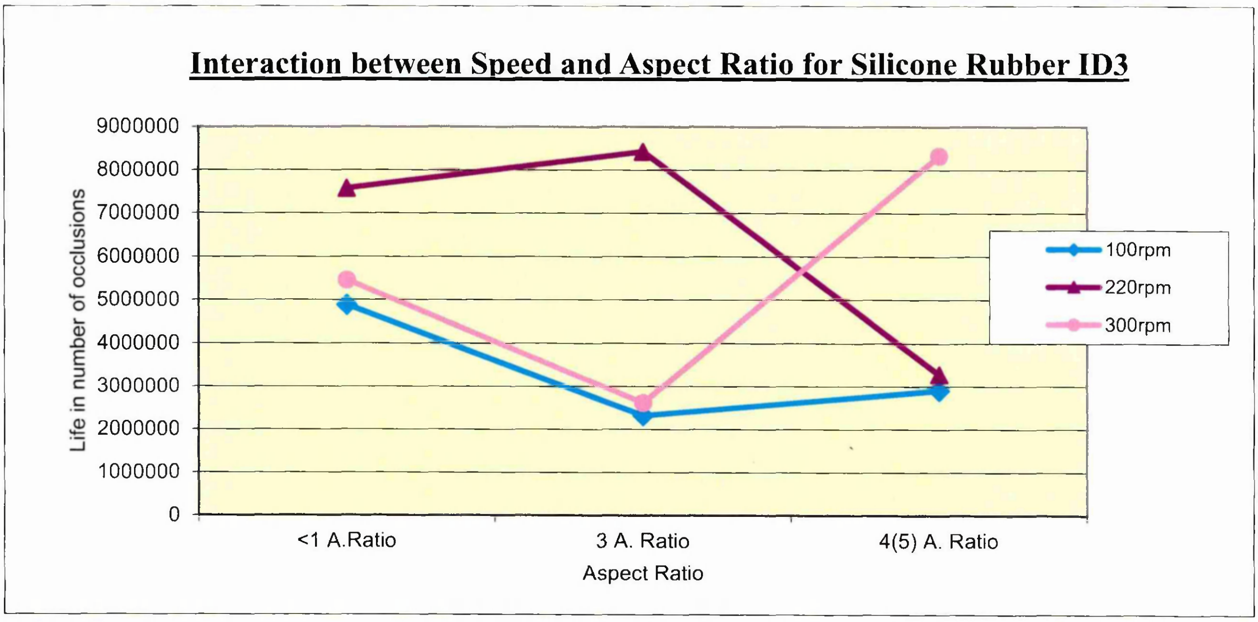
9.19 Appendix 19: Interaction between factors – Silicone Material 3








Interaction between Temperature and Wall thickness for Silicone Rubber ID3
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
Life in bumber of occlusions
4˚C
23˚C
40˚C
Temperature / ˚C
1.6mm Wall thickness
2.4mm Wall thickness


Interaction between Speed and Aspect Ratio for Silicone Rubber ID3
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
Life in number of occlusions
<1 A.Ratio
3 A. Ratio
4(5) A. Ratio
Aspect Ratio
100rpm
220rpm
300rpm

9.20 Appendix 20: Chemical Compatibility Tables for Silicone rubber and the thermoplastic rubber EPDM/PP blend (79)

CHEMICAL	SI	TPR
A		
Acetaldehyde	C	A
Acetic acid	A	B
Acetic acid anhydride	C	B
Acetone	B	A
Acetylene	-	A
Aluminum chloride solution	B	A
Aluminum sulphate solution	A	A
Ammonia (anhydrous)	A	B
Ammonium chloride solution	-	A
Ammonium hydroxide solution	-	A
Ammonium sulphate solution	A	A
Amylacetate	C	A
Amylalcohol	C	A
Aniline	-	B
Asphalt	C	C
ASTM Oil No. 1	B	C
ASTM Oil No. 3	C	C
ASTM Reference Fuel A	-	C
ASTM Reference Fuel B	-	C
ASTM Reference Fuel C	-	C
B		
Barium hydroxide	A	A
Beer	A	A
Benzaldehyde	C	B
Benzene	C	B
Benzene chloride	-	C
Benzol	C	C
Borax solution	-	A
Boric acid solution	A	A
Bromine liquid (anhydrous)	C	C
Butane	-	B
Butyl acetate	C	C
Butyraldehyde	-	B
Butyric acid	-	C
C		
Calcium bisulphate solution	A	B
Calcium chloride solution	A	A
Calcium hydroxide solution	A	A
Calcium hypochlorite solution (20%)	B	A
Calcium hypochlorite solution (5%)	B	A
Carbon dioxide	A	B
Carbon disulphide	-	B
Carbon monoxide	A	C
Carbon tetrachloride	C	C
Caustic potash (see potassium hydroxide)		
Caustic soda (see sodium hydroxide)		
Chlorine gas (dry)	-	C
Chlorine gas (moist)	-	C
Chloroacetic acid	C	A
Chlorobenzene	C	C
Chloroform	C	C
Chlorosulphonic acid	-	C

Explanation of abbreviations

A = resistant
B = moderately resistant
C = not suitable
- = untested

CHEMICAL	SI	TPR
Chromic acide (10-50%)	C	C
Citric acid solution	A	A
Cotton seed oil	A	A
Creosote	-	C
Cupric chloride solution	A	A
Cupric sulphate solution	A	A
Cyclohexane	C	C
D		
Dibutyl phthalate	-	A
Diethyl ether	-	C
Diethyl sebacate	-	B
Dioctyl phthalate	C	B
Dowtherm A	-	C
E		
Epichlorohydrin	-	B
Ethanol	A	A
Ether	-	C
Ethyl alcohol	A	A
Ethyl chloride	C	B
Ethylacetate	B	A
Ethylacetate dichloride	B	B
Ethylacetate glycol	A	A
Ethylacetate oxide	-	C
Exxon 2380 lubricating oil	-	C
F		
Ferric chloride solution	A	A
Fluorosilicic acid	C	B
Formaldehyde (40%)	-	A
Formic acid	B	A
FREON 11	C	C
FREON 12	C	B
FREON 22	C	C
FREON 113	-	C
FREON 114	-	C
Furfurol	-	B
Fyrquel 220 (hydraulic fluid)	-	-
G		
Glue	-	A
Glycerine (90%)	A	A
Grease	C	C
H		
n-hexane	C	C
Hydrazine (diamide)	C	A
Hydrochloric acid (20%)	B	B
Hydrochloric acid (37%)	B	A
Hydrofluoric acid (48%)	-	B
Hydrofluoric acid (75%)	-	C
Hydrofluoric acid (anhydrous)	-	C
Hydrogen	A	A
Hydrogen cyanide	-	A
Hydrogen peroxide (90%)	-	B
Hydrogen sulphide	-	A
J		
JP-4	-	C
JP-5	-	C
JP-6	-	C

CHEMICAL	SI	TPR
K		
Kerosene	C	C
L		
Lactic acid	A	A
Linseed oil	-	B
M		
Magnesium chloride solution	A	A
Magnesium hydroxide solution	-	A
Mercuric chloride solution	A	A
Mercury	A	A
Methanol	A	A
Methyl alcohol	A	A
Methylene chloride	-	B
Methylethyl ketone (MEK)	-	A
Mineral oil	A	C
Mobil XRM 206A	-	-
N		
Naphthalene	C	C
Naptha	C	C
Nitric acid (10%)	B	B
Nitric acid (30%)	B	B
Nitric acid (60%)	C	C
Nitric acid (70%)	C	C
Nitric acid (fuming)	C	C
Nitrobenzene	C	A
O		
Iso-octane	C	C
Oleic acid	-	B
Oleum (20-25%)	C	C
P		
Palmitic acid	-	B
Perchloroethylene	B	C
Phenol	C	B
Phosphoric acid (20%)	-	A
Phosphoric acid (60%)	C	A
Phosphoric acid (70%)	C	A
Phosphoric acid (85%)	C	A
Pickling solution 17% Nitric acid 4% Hydrofluoric acid	-	C
Pickling solution 20% Nitric acid 4% Hydrofluoric acid	-	C
Picrinic acid	C	B
Potassium dichromate solution	C	A
Potassium dichromate solution (dilute)	-	A
Iso-propyl alcohol	A	B
Iso-propyl ether	-	C
Pydraul 312C	-	C
Pyridine	C	B
Q		
QFI-2023 (Silicone brake fluid)	A	-
Quick silver (mercury)	A	A
R		
Ricinol (Ricinus oil)	A	B

CHEMICAL	SI	TPR
S		
SAE Oil No. 10	-	C
Sea water	-	A
Shell Turbine Oil 307	-	C
Silicone grease	C	A
Skydrol 500	B	A
Skylube 450	-	-
Soap solution	A	A
Sodium chloride solution	A	A
Sodium dichromate (20%)	-	A
Sodium hydroxide (20%)	B	A
Sodium hydroxide (46.5%)	B	A
Sodium hydroxide (50%)	B	A
Sodium hydroxide (73%)	B	A
Sodium hypochlorite (5%)	B	A
Sodium hypochlorite (20%)	B	A
Sodium peroxide solution	C	A
Soya bean oil	A	C
Stannic chloride	-	-
Stannic chloride (15%)	-	B
Steam	C	A
Stearic acid	A	B
Styrene	C	C
Sulphur (molten)	A	A
Sulphur dioxide gas	A	A
Sulphur dioxide liquid	-	A
Sulphur trioxide	B	B
Sulphuric acid (< 5%)	A	A
Sulphuric acid (5-10%)	A	A
Sulphuric acid (10-50%)	-	B
Sulphuric acid (50-80%)	-	C
Sulphuric acid (60%)	-	C
Sulphuric acid (90%)	-	C
Sulphuric acid (95%)	-	C
Sulphuric acid (fuming, 20% oleum)	C	C
Sulphurous acid	C	C
Sunoco XS-820 (EP Grease)	-	C
T		
Tannic acid	B	A
Tartaric acid	A	B
Tetrahydrofuran	C	C
Toluene	C	C
Tributylphosphate	-	C
Trichloroethylene	B	C
Tricresylphosphate	C	A
Triethanolamine	-	A
Trisodium phosphate solution	A	A
Tung oil	-	C
Turpentine	C	C
V		
Varnish	-	C
W		
Water	A	A
X		
Xylene	C	C
Z		
Zinc chloride solution	-	A

9.21 Appendix 21: Statistical method for determining reliability and confidence levels

Calculator for the number of test items required to demonstrate a given level of Reliability to a specified level of Confidence

The basic formula for nil failures is

$$n = \frac{\log (1-C)}{\log R}$$

where

n = number of items tested

C = level of Confidence (e.g. 0.95 which is 95%)

R = level of Reliability (e.g. 0.975 which is 97.5%)

CALCULATOR:

1 To calculate the number of test items required in order to demonstrate a given level of Reliability and Confidence (assuming nil failures)

Enter the level of Reliability specified	0.925	(between 0 and 1)
Enter the level of Confidence specified	0.95	(between 0 and 1)
Number of test items required =	**38.4**	

2 To calculate the level of Confidence from a given level of Reliability and an actual number of items tested (nil failures)

Enter the level of Reliability specified	0.96	(between 0 and 1)
Enter the number of items tested	80	
Level of Confidence demonstrated =	**0.961832**	

3 To calculate the level of Reliability from a given level of Confidence and an actual number of items tested (nil failures)

Enter the level of Confidence specified	0.95	(between 0 and 1)
Enter the number of items tested	80	
Level of Reliability demonstrated =	**0.963246**	

4 Calculator in the event of more than zero failures

Enter the level of Reliability specified	0.925	(between 0 and 1)
Enter the number of items tested	80	

No.of Failures (n)	Probability of (n) failures	Cumulative Probability	Level of Confidence demonstrated
0	0.0020	0.0020	**0.998044**
1	0.0127	0.0146	**0.985357**
2	0.0406	0.0553	**0.944726**
3	0.0857	0.1409	**0.859070**
4	0.1337	0.2746	**0.725377**
5	0.1648	0.4394	**0.560610**
6	0.1670	0.6064	**0.393616**
7	0.1431	0.7495	**0.250478**
8	0.1059	0.8554	**0.144576**
9	0.0687	0.9241	**0.075882**
10	0.0395	0.9637	**0.036337**
11	0.0204	0.9841	**0.015933**
12	0.0095	0.9936	**0.006420**
13	0.0040	0.9976	**0.002386**
14	0.0016	0.9992	**0.000820**
15	0.0006	0.9997	**0.000262**
16	0.0002	0.9999	**0.000078**
17	0.0001	1.0000	**0.000022**
18	0.0000	1.0000	**0.000006**
19	0.0000	1.0000	**0.000001**
20	0.0000	1.0000	**0.000000**

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